SYNTHESIS REPORT

FOR PUBLICATION

CONTRACT No :BRE2-CT92-0314

PROJECT No : BE 5880

TITLE : Innovative constituents for improving the compressive strength of composites
(ICOMP)

PROJECT
COOIUXNATOR : DASSAULT AVIATION

PARTNERS :
- SNPE
- DAIMLER-BENZ
- DLR
- DRA
- NLR
- (_)NE~
- AEROSPATIALE
- IJJIVERSITY OF PORTO

ST.ARTING DATE :1 FEB 1993

DURATION :42 MONTHS

PROJECT FUNDED BY THE EUROPEAN
COMMISSION UNDER THE 13RITE/EURAM
PROGRAMME

ICOMP

Innovative constituents for improving the compressive strength of composites

Synthesis report

ABSTRACT

Beside a constant increase in carbon fibres tensile properties, the compressive strength of composite has not been improved since their introduction in aircrafl primary structures, In order to take full advantage of the second generation Intermediate Modulus carbon fibres, a target increase of 300/o in composite compression is necessary to recover a more symetric behaviour, as most applications are subjected to alternative tension and compression.

This progmrnme has contributed to understand various composites compressive failure modes, and to address quantitative requirements on identified key constituents. Experimental and theoretical developments have demons&ated that compression is cIearly mati dominated for composites made out of low inertia tibres such as High Strength and Intermediate Modulus carbon or such as glass. The 300/0 target has been showed to be directly dependent of an improved neat resin shear bebaviour, by increasing preferentially the yielding strength, but also the modulus. The fibre~matrix interface shear load transfer ability appears to be of second order for compression. The initial fibres misalignment was also identified as an important parameter, which controls the fikiments micro-buckling phenomenon. However, ICOMP experimental trials to install pre-tension in plies are not seen as industrially applicable. For designs driven by compression, the recommendations are to use product forms which minimize fibres waviness.

Therefore, propckitions of innovative materials have been concentrated on new matrices development. As po@rner experts have stated that the organic chemistry has probably reached an absolute limit with respect to shea properties, the introduction of fme particles in tough resins was investigated to match the shear behaviour requirements. An encourageous demonstration has been performed using a low class ductile polymer system. The addition of whiskers into the matrix increased both the neat resin smength and moduius, which enhanced the unidhectiomd composite compression in the forecast proportion. The lack of pax-tides and resti optimisation, within ICOMP time frame, did not Iead to an improved high class aeronautical material. However, this innovative route appears to be the only solution to combine prepregs processability, material cost and compression improvement. Furethemore, experimental indications suggested that this gain will directly be tmnsposed into design criteria, such as compression after impact or compression of laminates containing a boit, withou degrading tensile properties and delamination resistance.

Dedicated inspection methods for in-situ damage monitoring or initial geometrical imperfections assessment were developed. A reliable compression tes was a key preliminary request, which led to propose an innovative procedure to measure intrinsic unidirectiona laminate compressive propefiies. The tremendeo~ improvement brought by the new specimen has already been taken into account for fiiture standards.

Summarizing the above, it can be concluded that this cooperation has been a unique opportunity to generate an homogeneous data basis, which made avai~abie for the fmt time quantitative requirements to improve composite compression. A promising solution has been investigate~ but it will require further research effort. One of the major benefit of this Brite/Euram project was to drastically decrease the iteration time and cost for a material development with detailed specifications from the end users. It has contributed *to* reoriente some research priorities for more composite introduction efficiency.

1- INTRODUCTION

1.1 General problem statement

In spite of a constant increase in the use of carbon fibre-epoxy matrix composites in advanced structures over the Iast I 5 years, their low compressive properties is one of the main mechanical limitations for a mote widespread integration of composites in vital structures of **any** industial field.

The recent development of Intermediate Modulus carbon fibres Ied to very high specific tensile strength of composites, with a gain of 50 **'/0** over the fust generation of High Strength fibres. Unfortunately, compressive propeflies, which *are* mainly governed by the matrix, did not improve at all.

Most of the complex structures are subjected to alternate tension and compression loads at an equivalent stres level (robots arms, boats mats and keel, aircraft fms). Because of the disymetric behaviour between tension and compression, it is not possible to take advantage of the very high properties in tension, as the compressive loading becomes the design limitation (see figure 1 which gives data for 60 **'%. volume** fibre content and a typical 180"C cure semi-toughened epoxy).

	0“ strength (MP@	0“ strength (MPa)	Approximate year of availability
~ (High Strength carbon fibrel I	I700	1600	197CI
(Intermediate modulus carbon fibre)	2600	1600	1985

Figure 1: Evolution of the composites tensi}eicompressive strength baiance

1.2 Programme objectives

As no improvement in compression has been noticed over the years, the main objective was to dearly identify the governing phenomen% in order to express quantitative requirements on constituents. By obtaining specifications for the fwst time, pilot composites were deveIopped to validate material concepts in order to increase the long flbres reinforced plastics compressive strength. A target of 30 % was set for carbon based composites, in order to approach a symmetric behavicwr between tension and compression for the new generation of fibres,

The fiie understanding of composite compressive failures has led to the following innovations and developments :

1 ReIiabIe compression testing procedure to assess intrinsic unidirectional strength of composites

2 Constituents and imperfections characterisation technics such as compressive properties of fibres, 3D pattern of initial fibre misalignment, damage growth under compressive loads.

3 Constitution of a unique data base in order to face a wide range of compressive failure modes, depending of the reinforcement ~aramide, glass, carbon high resistance, carbon high moduIus, Boron) seIected according to the industrial applications.

4 Development of a simulation tool for key constituents properties specifications.

5 Identification on key constituents evaluations to reduce time and cost of composite development and characterisation.

This project complied with the aims and objectives of the Brite,Qurarn programme by increasing the competitiveness of the European aeronautical industries, institute and materials producer.

This is a key economical and industrial opportunity to reduce the time and cost of materials qualification, by forcing the dialogue between the material supplier and the early requirements of the end users. The indust~ will not afford any longer to pay the development and characterisation of dozens of materials, for fmaiy selecting one for application. Of course, this relies on abetter specifications by the end users.

Even if the project focuses orI aeronautical applications, the compressive weakness is common to a wide variety of Iong tibres composites. Furthermore, several industrial sectors will get the benefits of this research, through the material supplier, who touches a wide breadth of composites applications [sporting goods, trains, boats, automotive, robots, *aircraft...). Also, the* research institutes invoked in this project are used to material and design problems coming horn all kinds of industrial sectors, and therefore they will contribute to the know-how difli.sion.

- For the specific case of aircrafi industry, weight savings is always a target, as it can reduce the Direct Operating Cost for wide body aircraft and enhance the flight range of a business jet. Another important issue for composites t%rt.her introduction being their costs, it may be time to slow down the development of expensive carbon fibres with better and better tensile properties as the design limitation is often on the compressive side, which is matrix dominated.

L3 Compression improvement issue for designers

For wingbox types of primary aircraft structures, the composite materials selection in terms of static strength to faihtre can be summarized as shown on figure 2. CormnerciaI composite, as avaiIable today, vary by carbon fibres smength class and by resin toughness. As suggested on figure 3, they are almost equivalent in compression. T%e remaining compromise lies between tensiIe properties and falling weight impact resistance. The properties reported on the **3 axes** of figure 2 used specimens and loadings which are descriptive of the most critica[desi~ requirements.

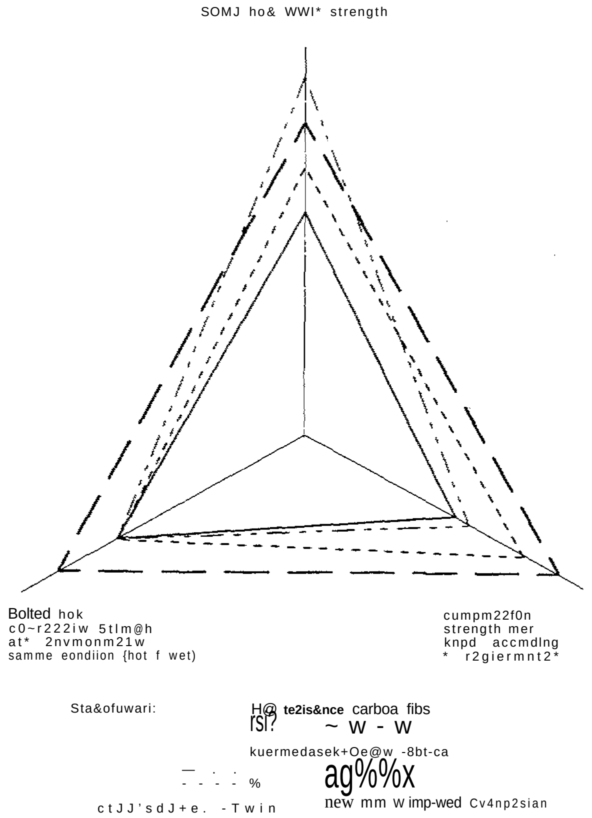


Figure 2 : Summary of key iaminates properties for aircrafl structural application

The compressive strength design limitation come from two aspects :
- Stress concentration around a bolt (FHC : Filled Hole Compression)
- Residual strength after falling weight (CAI : Compression After Impact}.

In the case of a wingbox with a low relative thickness, as for modern fighters (1), the compression strength criteria are dimensioning for about 15 ‘A of the composite structural weight. The main design criteria is iinked to rigidity aspects (flutter and aeroelasticity). The remaining factors are tension with bolts and bearing problems.

In the case of a wingbox with high reiative thickness, as civil application as ATR commuter (2), the upper skin design is much more induced by both compressive strength aspects. For this type of application, a gain of intrinsic composite compressive performances will lead to significative weight savings.

The curve with circles on figure 4 suggests that a gain in compressive properties will permit, in a conservative marmer with respect to any impact threat reglementation, to realize some weight savings for are subjected to compressive loads (3).

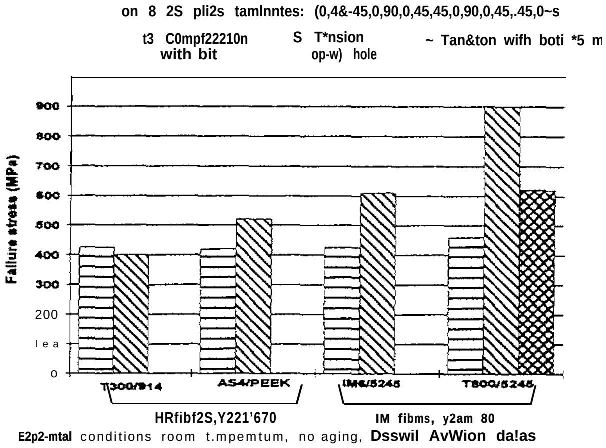


Figure 3: Tension vs compression of standard laminates containing a bolt

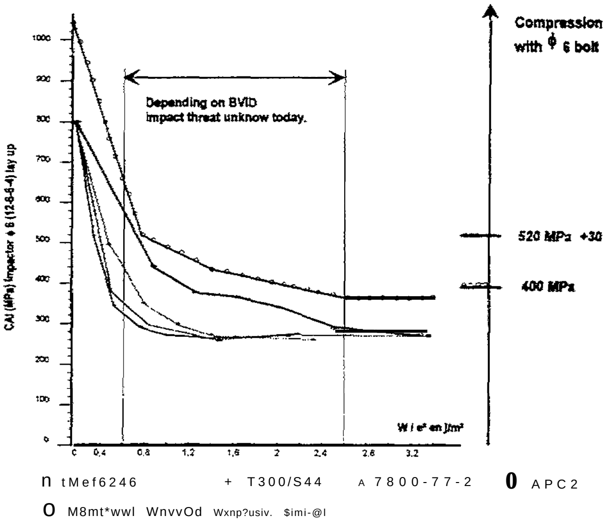


Figure 4: Compression after impact and bolted hole compression

2- MEANS TO ACHIEVE THE OBJECTIVES

2.1 Consortium **of pwt~ers**

The multidisciplinary aspects of the research were covered by complementary partners:

Aircraft and vehicules manufacturers mainly specified detailed requirements, manufactured the laminates with their usual industrial equipments, performed some complex tests and run some calculations:

- Dassault Aviation (France) - [Coordinator]
- A6rospatiale (France)
- Daimler Benz (Germany)

Research institutes brought their experience about materials physico chemical knowledge, mechanical testing, characterisation of geometrical imperfections and scientific computations:

- DRA (United Kingdom)
- ONERA (France) - [associated to Adrospatiale]
- DLR (Germany) - [associated to Daimler Benz]
- NIX (I?etherkmds)
- U. of Porto (Portugal) - [associated to Dassauk]

A continuous dialogue was engaged with the material supplier who had the expertise to develop prototype resins and to manufacture prepregs. Also here, using industrial prepregging lines forced to take into account resins rheology and processabildy cons~aints to raise all the questions to obtain an industrial product.

- SNPE (France).

2.2 **Project technical approach**

The figure 5 presents the project flow diagram (10).

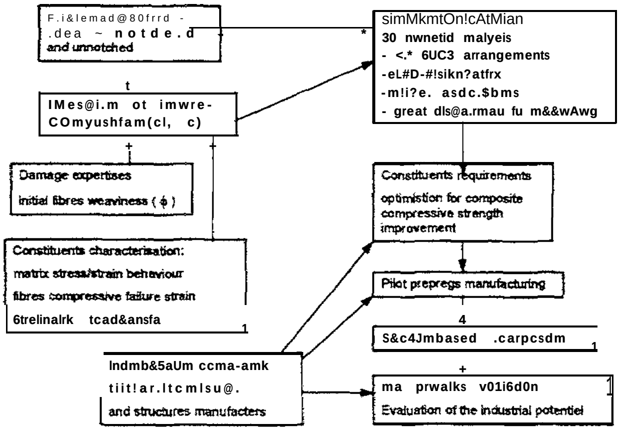


Figure 5: Project flow diagram

Task 1:

Development of a simulation tool in order to quantify the relative et%ect of the many material parameters that influence the composites compressive strength. Only a numerical computation could reproduce a realistic fibres arrangement in the laminates and deal with elasto-plastic matrix behaviour.

In order to validate the model on a wide variety of compressive failure scenarios, the key point was to generate an experimental date base which systematically gives :

- Constituents medxmica~ behaviors.
- Initial geometrical imperfections within the laminates (fibres misalignment).
- Composites compressive response.

Several composites fkmiiies were elaborated and studied, through various reinforcement types and through prototype resins with different shear behaviors in order to find out the governing parameters.

Task 2:

Synthesis governing parameters, specifications of constituents properties to improve composite compressive smength, proposed materials answers taking into account processability (prepregging and laminates molding).

Task 3:

Development of pilot prepregs from specifications and innovative constituents to increase compressive properties. Elementary evaluations with respect to unidirectional compressive properties.

Task 4:

Characterisation of general properties on more complex laminates. Synthesis and conclusions to reach an industrial material with improved compressive performances.

3- OVERVIEW OF RESULTS

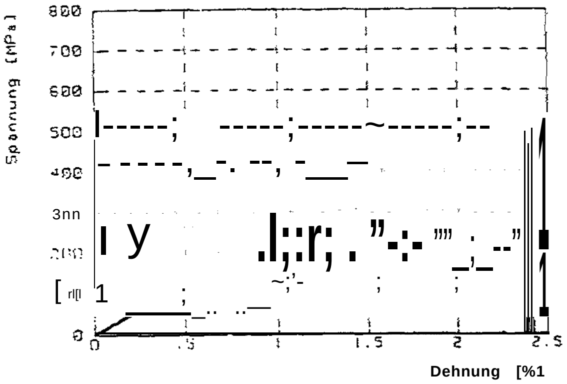
3.1 Unidirectional compressive failure understanding and simulation (Task 1)

Develo~ment of a new corm)ression test

Prior to ICOMP, a round robin test between the research institutes involved in the project (4), demonstrated that all the standard tests used to measure an unidirectional compressive strength were not satisfactory at **all.**

Furthermore, there was evidence of much higher compressive strain to failure, monitored during flexure tests or during a compressive test with the 0° plies being protected by aramid fabrics. This suggested that the intrinsic 0° compressive strength was not measured. Of course, it was of a great importance for the consortium to dispose of a reliable test to evaluate composite compressive properties.

A multiangle laminate was selected, and 0° ply response is computed as shown in figure 6. For the reference material (carbon T300/DA 508 epoxy), 0° ply strain the failure of- 2,5 % have been measured, compared to - 1,5 % currently reported in the litterature.



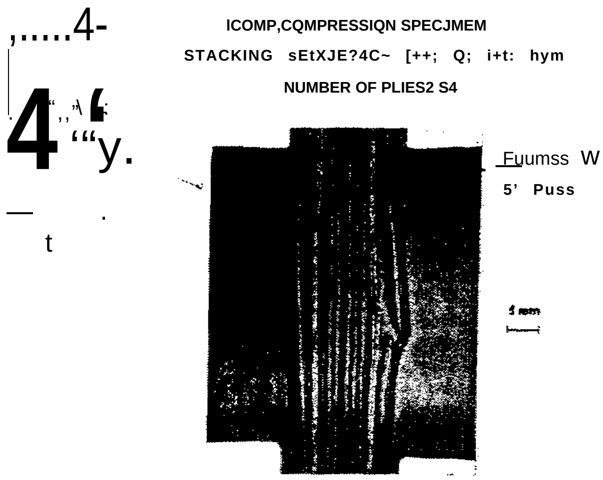


Figure 6: Result of unidirectional compressive behaviour with the new testing procedure

A reliable compressive test has been successfully developed. It consists of a (f60a, O_z, ~60Js laminate, without tabs, loaded in shear with a celanese rig.

Fibra'matrix cmmles selected and tests momanrne

From the background on compressive failure modes of composites (5) summarized in figure 8, the following fibre/matrix combinations have been tested to get the various fracture responses {see figure 7].

The proposed experimental data base construction was aimed to study the following characteristics, that were always mentioned in the literature as influencing the compressive response, but without any quantitative and hierarchy precision :

- matrix stresshtrain behaviour
- geometrical imperfections (initial fibres misalignment)
- fibre/ma&ix interface
- fibres properties
- fibres volumic content.

code	Fibre	Resin
1T	r300 carbon from Toray	DA508 reference commercia epoxy from SNPE with 60 % fibre volumic content
1P	T300 without surface treatment	DA508
1T'	T300	DA508 with -8 % of fibre volume content
1T"	T300	DA508 with + 14% of fibre volume content
1V	E-Glass horn Vetrotex	DA508
1X	M40B, high modulus cm-bon fibre tlom ToraY	DA508
1Y	M6013, ukra hi~ modulus carbon fibre from Torav	DA508
1W	3oron tiom Avco	DA508
2T	moo	Experimental resin 2, non stoechiometric epoxy amine system with small amount o CTBN rubber
3T	T300	Experimental resin 3: simpl epoxy amine system
4T	'r300	Experimental resin 4: low modulus resin di and t functional epoxy, amine hardener, him ratio of CTB
5T	T300	Experimental resin.5 : same 'as resin 4 + 5,9 ?40 of whiskers volumic content
6T	T300	Experimental resin 6: simple epoxy amine system
2V	E-Glass	Experimental resin 2
3V	E-Glass	Exuerimentzd resin 3
4V	E-Glass	Experimental resin 4
5V	E-Giass	Exoerimenta~ resin 5
6V	E-Gkss	Experimental resin 6

Figure 7: Selected fibre/matrix couples.

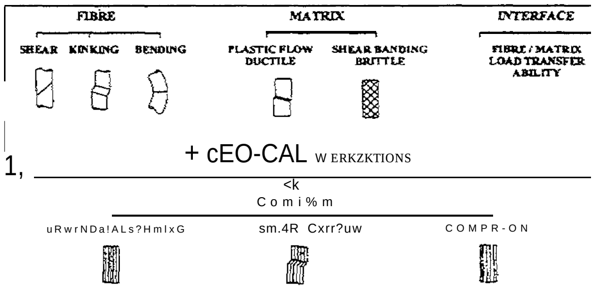


Figure 8: composites compression failure modes

in the case of the mostly used reinforcements Glass or high resistance amd irttermediate modulus carbon, the scientific community agreed to ciass the compressive failure as matrix dominated, exhibiting fibres micro buckling. The choices of experimental matrices required a lot of work, because mechanical behaviors requirements was adressed to SNPE, in order to fmd out if the key parameter was more the matrix rigidity or yielding strength. Of course these resins were also to be processable to impregnate a tape of 60 % fibres content. Beside all the physico chemical evaluations and preliminary composites characterisations on a simple interlaminar shear test SNPE selected five experimental resins with distinguished stressktrain behaviors presented in figure 9.

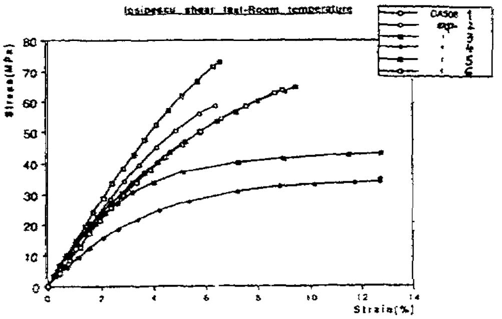


Figure 9: Resins shear behaviors at room temp.

The figure 10 describes the tests matrix that has been performed. It is important to notice the following points:

- This unique data base was rich because of systematic and parallel evaluations of composites (subtask 1.1), of constituents (subtask 1.3), of initial imperfection and of failure expertise (subtask 1.2).

- Various testing temperatures were used to simply get a wide range of matrix stress/strain behaviors without affecting the reinforcements properties.

	Fibre / Matrix	Boron from AVCO W	HMcarbon M40 from TORAY X	HMcarbon M60 from TORAY Y	HRcarbon T300 from SOFICAR T	EGlass from VETROTEX V	T300 poor interface P
1	Reference matrix DA 508 SNPE	C I F } HT	C I F } HT	C I F	C M I F } HT	C I F } HT	C I } HT
2 3 4 5 6	5 experimental resins with different G/S ratios (SNPE)				C M I } HT	C	
I	1st variation of fibre volume content (about 50 %)				C		
J	2nd variation of fibre volume content (about 65 %)				C		

C : Tests on composites (subtask 1.1)

- (f60z, 0_1, $+60_3$)~ compression ; 0° ply response computation by (f60) behaviour deduction
- (*60)$_1$0 compression (added with respect to the technical annex programme)
- 90” compression to get a resin dominated indicator including the interface aspects
- * 45° in tension and compression to get a resin dominated indicator inciuding the interface aspect.
- Short Beam Shear test on 0° kuminate to get a resin and interfaee dominated indicator
- Initial tlbres wea~iness measurement (subtask 1,2).

M : Direct matrix stresshtrain behaviour (subtask 1.3) tensile and shear (Iosipescu) tests.

I : Fibre/matrix interface and load transfer capaci~ (subtask 1.3) Pull out and fiagementation tests.

F: Fibres compressive properties (subtask 1.3) loop tests and compression on 1 embedded filament.

HT: Testing temperatures: 20,70, 120, 140°C.

Other supports provided by subtask 1.2:
- Acoustic Emission during composites loading to detect fust damage stress Ievel.
- Fractographies of compressive faihtres.
- Compressionfbending tests inside a scanning electron microscope.
- Development of a technic to assess the initial fibres misalignment.

Figure 10: Summary of experimental investigation

Facts rrrovided bv theoritica[artalvsis

Most of the theoretical studies achieved these ten last years tend to prove that the analysis performed by 13udiansky in 1983 is particularly relevant. The main results of this analysis is the following :

$$\sigma_{11C} = \frac{G_{12}}{1 + \varnothing \frac{G_{12}}{\tau y}}$$

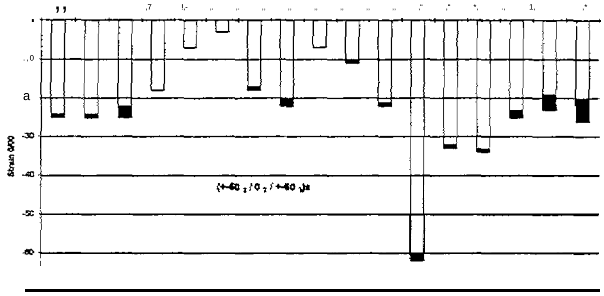
where al ~$_c$ is the compressive failure stress, where G ~$_z$ is the UD shear modulus, where 0 is a characteristic of the fiber waviness and where 7y is the shear yield stress.

The frost job was to confm the Budinasky’s relationship by a 3D numerical computation, taking into account all the detailed non linear behaviors of constituents and real initial imperfections. In the case of most composite materials, where compressive failure is governed by fibre microbuckling, a 3D finite element model has been developed (6). The ultimate goal was to obtain a simulation tool, but a preliminary objective was to help to quantify the relative influence of fibres misalignment, resin modulus and resin yield strength.

Ran~e of comDressive failure modes

The figure 11 reports the 0° ply compressive failure strains of the various composites. These results were obtained with the new test procedure on (MOz,Oz, f6UJ~ laminates. Looking **at the** measured strain to failure of neat fibres, it appears clearly that boron and high modulus carbon fibres ultimate strains pilot thelaminates faiiure. Fratographies confwed the ruine modes.

Standard carbon fibres and Glass reinforcements based composites are relevant of microbuckling failure mode, with all the resins studied. Boron or high moduius carbon fibres based composites failed by filaments compression, with regards to their higher inertia.



Fibre	~ Code I	Strain range at faiIure ('%)
T300	T	3.95-4.61 I
E-GIass	v	>9,14
M40	x	1,07-1,65
Boron	w	2.20-2,81

Figure 11: Compressive strain to faiIure of composites and fibres

Focusing on carbon and Glass based composites, which are the mostly used reinforcements in industrial products, the figures 12 and 13 show strong correlations between composite shear values and 0° iaminate compressive strength a ~ ~$_c$. The shear properties G ~$_z$ and T$_{,z}$ were deduced tlom (k 45°) tensile and compressive tests.

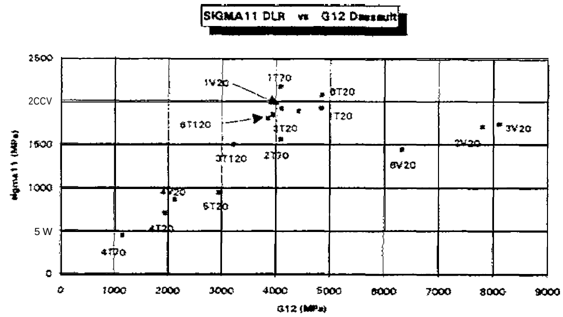


Figure 12: Correlation between cr$_{(}$ ~ and G$_{,z}$.

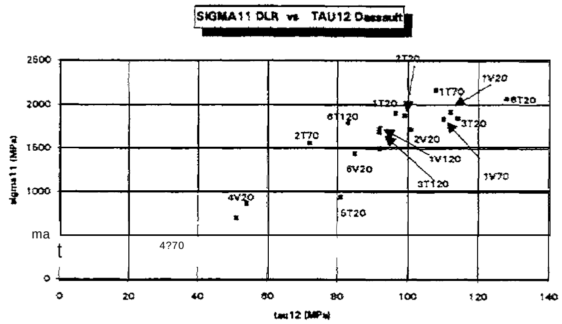


Figure 13: Correlation between 6$_{11}$ and T$_{lz}$.

The important point to notice, is that G$_{lz}$, T$_{lz}$, or G$_{=}$ are laminate properties directly governed by the matrix. They have to be thought as future good elementary evaluations as they also include the fibre/matrix interface mechanical response.

By recalling that for each fibre/mahix couples, tests were performed under temperatures that only affected the resin behaviors, the following statement is experimental confined :

0° compressive failures for Glass and Carbon composites are matrix dominated.

Govemirw Darameters and simulation tool

The influence of the parameter variation on the unidirectional compressive strength al ~ can be analysed by applying the logarithmic derivation to the Budianski's relationship :

$$\frac{d\sigma_{11C}}{\sigma_{11C}} = \frac{\sigma_{11C}}{G_{12}}\,\frac{dG_{12}}{G_{12}} + \left(1 - \frac{\sigma_{11C}}{G_{12}}\right)\left(\frac{d\tau}{\tau} - \frac{d\varnothing}{\varnothing}\right)$$

The mean value of the experimental ratio al JG$_{,2}$ for all carbon and Glass/matrix combinations is around 0,4. Therefore, the equation 1 precises that an improvement of al $_1$ can be obtained by an increase of G_{12} and the resin yield strength r, or a decrease of the initial fibres misalignment 0 (7).

Equation 1:

$$q \;=\; \sim_4 d\ G_{12}$$

$$\sim l \sim c \quad {}^{7} \sim + \;"\; ' \;[\; ? - \% \;?$$

In order to describe the initial fibres misalignment by a simple parameter 0, it was essential to develop a technic to characterize the real fibres distribution in rhe cured kuninates. The methodology consists in petiorming several cross sections, polishing them and by data processing (8) to reconstruct the fibres path as illustrated in figure 14. The deviation angles distribution is homogeneous through the ply thickness, wherever the ply position through the laminate thickness.

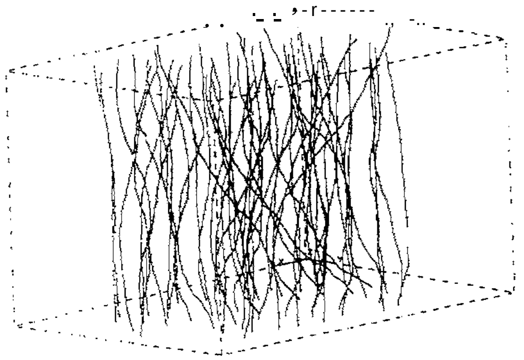


Figure 14: Initial tibres waviness for IT (T300/DA508) material

The fibres misalignment data are presented in the form of standard deviations of data fitted centred normal distributions for both in-plane and out-of-plane (through-the-thickness) misalignment angles. Only the in-plane misalignment data are considered since the developed models assume failure occurs by in-plane microbuckIing. The fibre misalignments are being simulated by a sine ctuwe with alternate ‘positive and negative angles. Therefore the sine curve characteristic angIe was taken as the average angle of each half normal distribution, i.e. :

$$0 = !^{*} Xf(X)\, dx$$

where :

$$f(x) = \frac{1}{c\ d\ \%} e\text{-}(x/5)v2$$

is the centred normal distribution density function.

The figure 15 gives the laminate input data for th evaluation of the iamina longitudinal compressio strength model. 7

Material \ Fibre ~ (code)		Resin ~	Vf	0 (deg.)
1P	T300	DA 508	0,606	1,4737
1T	T300	DA 508	0,574	0,7859
1T'	T300	DA 508	0,546	0,76
i-r’	T300	DA 508	0;672	0,9012
2T	T300	exp.2	0,639	0,9128
3T	T300	exp.3	0,605	0,7548
4T	T300	exp.4	0,457	0,8186
ST	T300	exp.5	0,499	0,8569
6T	T300	exp.6	0,598	0,8282
lV	E-Glass	DA 508	0,539	0,5888
I w	Boron	DA 508	0,66	0,1835

Figure 15: Characteristic angle for initiaI fibres misalignment description

All the input being available, the 3D model appears t be globally in good agreement with the experimenta data as suggested on figure 16.

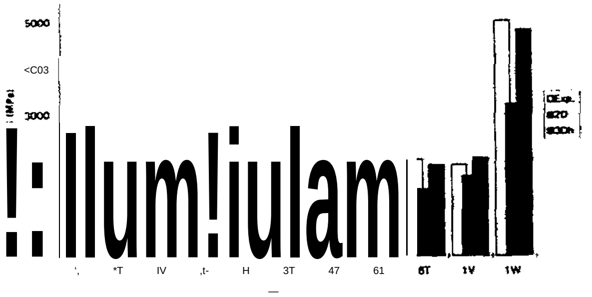


Figure 16: 3D model prediction and experimental compressive smength data

A 3D simulation tooI has been successfully developed.

A elasto-plastic behaviour of the matxix and a fibre misaligmnent characteristic angle permits to identify th key constituents parameters.

The unidirectional composite compressive strength ca be improved mostly by increasing the resin yiel strength and by decreasing the initiaI fibres waviness and secondly by increasing the unidirectional laminat shear modulus.

As all the carbon and Glass molded laminates exhibite fibres waviness in the same range (9s 0,80), the mos effective variation is seen to be given by a improvement of the resin yield stress.

A precious indication that tends to corroborate this philosophy can be found in the following idea : since it is clearly reasonnab!e to admit that q, (a resin property) and ~$_{12}$(a composite property) are closely linked, it is interesting to evaluate the link between 0$_{1}$,$_{C}$and ‘r$_{IZ}$to be sure that the phenomenon is well understood.

The main results issued t?om task 1 are presented in figure 17. They are self explanatory by concluding that the improvement of ~$_{Y}$(resin property) therefore the improvement of ?$_{IZ}$(composite property) leads to an increase of cr, ~$_{c}$, which is the desired result.

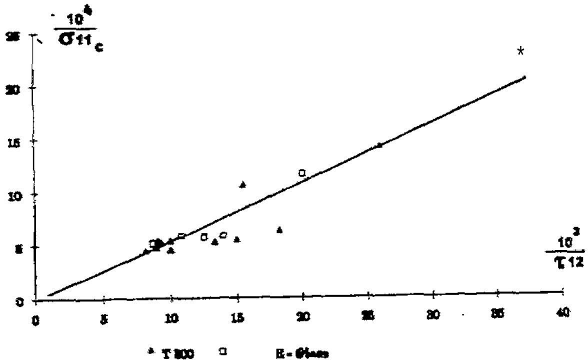


Figure 17 : Correlation between σ_{11} and τ_{12}.

Ex~erirnental materials indications

As shown on figure 16, except the boron laminates, non of the experimental resins associated to carbon fibres enhanced better performance than the reference 1 T (T300/DA508) commercial product from ShPE.. It is recalled that at this stage, the main goal was to get experimental evidence of governing constituents parameter, as successfully demonsmated in the previous chapters. According to resin stress/strain behaviors plotted in figure 10, this resuIt is logic because neither the modulus nor the yield strength of any experimental matrix was higher than the reference material. However, the following other indications are important to notice :

- The fibre/matrix interface importance, from the mechanical point of view, has been assessed throughout the tests campaign. The load h-ansfer ability has been measured by fragmentation or pull out technics. With fragmentation shear txansfer values varying from 32 to 60 MPa, no evidence of any interracial problems have been pointed out in composite compression failures. Furthermore, the 3 D simulation, which considered the interface as a perfect joint, matched all the experimental results (see figure 16). Even material 1P, directly comparable with the reference 1 T but elaborated with untreated carbon fibre, did not failed in compression from an interracial problem. The fibre~matrix shear load transfer is seen as a secondary property for composite compression (based on the range of materials tested in iCOMP).

- Acoustic Emission during the compressive loading of the (i60$_{2}$, O$_{z}$, +60Js specimens and ultrasonic inspections at various stress levels are valuable tools to help the detection of degradation stress levels.

- A compression-bending test campaign has been performed inside a Scanning Electron Microscope. FaiIures have been confined to be matrix dominate~ inducing fibres micro-buckling. With a grid deposition technics, local strain to failure in the range of 2 to 2,5 ‘A were observed for material 1 T. This was a conflation of ultimate 0° compressive strain measured with the ICOMP new test procedure.

- From a resin formulation point of view, the case of experimental matrices 4 and 5 is very interesting. Dealing with a very ductile resin 4, the introduction of 5,9 volumic percent of whiskers had the effect of increasing both the modulus and the shear yield strength, resulting in experimental resin 5. The corresponding O“ compressive strength of the 5T composite increased by 33 % over the 4T value. This result was obtained with resin 4, which had very low properties and which led to poor laminates soundness. Nevertheless, this axis of resin improvement for increasing composite compressive strength was experirnentaly demonstrated for the fmt time. preliminary trials done with SNPE, adding particles in the reference resin 1, were not successful, as only the modulus was increased, while, because of the resin brittleness, the shear yielding strength was severely decreased. Figure 18 summarized the impact of resin behaviors on 0° compressive strength. Adding particles to a ductile matrix seems to be an axis for composites compression improvement.

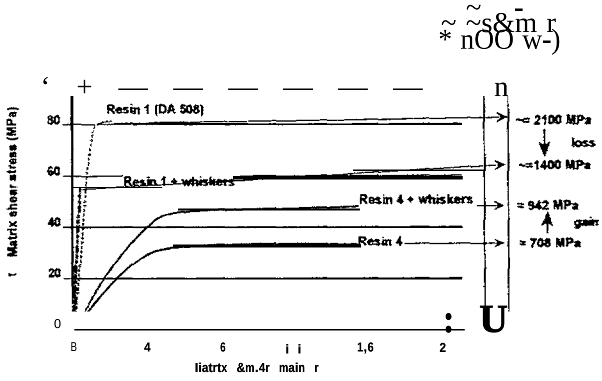


Figure 18: Resin+ particles effect on 0° composite compressive strength

- Part of subtask 1.4, an attempt to get an experimental verification of initiai fibres misalignment has been introduced during the project progress. An experimental autoclave tooi has been manufactured to cure the laminates with installed pretension in the 0“ piies of the multi-angies [ay-ups. 3 trials with increasing pre-tension load have been performed.

  The values of compression strength had increased of 4 °/o and 7,S °/o respectively to stacking sequence 32 plies [(+/-60z/Oz/(+/-6)2)OzO(+(60)2]S2]S ~d 32 plies quasi-istropic [(Oi451-45/90)& after 800Kg pre-tension. Direct waviness measurements confined also improvement of fiber alignment. The acoustic emission activity was reduced in the case of pre-tensiied specimens, suggesting less microbuckling phenomena.

The increase of pretension values tends to improve the composite compression.

The other main objectives of subtask 1.4 were to move to muki-angIes laminates in order to get complementary mechanical indications such as :

. Clustering effect of 0° plies.
. Hole stress concentration in isotropic laminate.
. Bolt torque effect.
. Delamination and impact resistance.,

Main conclusions on multi-andes laminates :

- Testing of (0°, ~ 60”) type laminates in the modified Celanese test rig resulted in high failure strains above 2 Yo. Clustering of 0° layers (tlom 1 by 1 ply, up to 4 by 4 plies) slightly reduced the compressive strength.

- The effect of clustering was more significant for quasi-isotropic laminates.

- No effect of clustering was observed for specimen cut out of laminate at a 22..5° angle.

- Clustering slightly increased the strength values during open hole tension and compression tests on quasi-isotropic laminates. As in tension, a damage growth mechanism (early spiitting of 0° layers and delamination) lowers the stress concentration factor for lay-ups with delamination tendencies. Testing of 22.5° specimens showed the reverse influence of clustering 0° pIies.

- For stacking sequences not delamination sensitive, ply 1 by 1, no significant damage have been monitored up lo 99?40 of the specimen collapse. For such industrial lay-ups which minimize edge sensitivity, open hole compression is governed by 0° plies failures.

- The applicability of a Point stress faihwe criterion to open hole compressive strength of quasi-isotropic laminates (subtask 1.5) involves the same cautions than for tension : inadequate for off axis loadings, and for strongly stacking sequence dependent strengths.

- FilIed hole compression on quasi-isotropic laminates showed the highest strength values for the i T material with the lowest degree of clustering. In all cases, the application of a torque on the bold slightly increased the compression strength (+6% for IT material and 1 by 1 ply sequence). This suggests that no major damage process is invo[ved during bolted hole compression with sequence insensitive to delamination.

- Compression after impact and mode H enerefg release rate were equivalent for materials IT and 6T. The bad quality of material 2T gave poor damage resistance under falling weight impact.

3.2 **Constituents requirements**

sylthesis of ~ovemirw Mrameters

The validi~ of equation 1, which summarized the governing parameters and their relative impact to increase the unidirectional composite compressive smength C_I*c, has been validated by task 1 experimental investigations and 3 D simulation.

Equation 1:

$$\frac{d\sim llc}{\sim llc} = 0,4 \; d: \; \sim \; {}^{+} \, ” \; d \; : \; - \; \% \; ?$$

with : $G_{[2}$ = unidirectional shear modulus
z = matrix shear yielding strength
0 = characteristic initial fibre misalignment.

~ ent (task 2)

According to task 1, the following leading ideas are driving the axis of improvement:

- Boron fibres were the unique obvious solution to increase the 0° compressive strength. However, *this* route is not selected for the following reasons :
  . Prepregs too stiff to lay on contours.
  . Toxic problems in the composite shop.
  . Unreasonable cost.
  . USA product, close to disappear.

- Focusing on carbon based composite, but philosophy also applicable to Glass, the least initial fibres waviness do enhance the compression *strength.* However, the pre-tension trials successfully performed with a complex autoclave tool concept seems not realistic in an industrial context on large contoured parts with complex lay-ups and ply drop-Offs.

  As the minimisation of fibres misalignment is not at the prepregs manufacturing level, but during the composite part moiding, this aspect is not taken into account for innovative constituents for unidirectional tape prepregs development.

- Following task 1 conclusions, figure 19 suggests the required neat resin shear behaviour, which should lead to a 30 ?40 gain on composite compressive strength (9), It is recalled, that all commercially available organic matices used in aeronautic structures, have almost the same shear yielding stress level, close to 80 ?vll% (see figure 20}.

  A gain of 30 ?40 on the neat resin shear yielding strength should lead to an increase of the same order for 0 compression, either for a T300 or a T800 fibre type, as the faih.tre mode is clearly matrix dominated. In the case of T800 type of reinforcement, the 0° tensile and compressive behaviors become almost symmetrical.

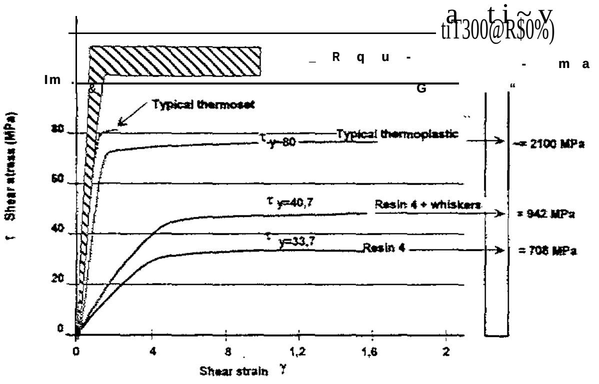


Figure 19: Neat resin behaviour requirement.

Previously to ICOMP project, all the work on matrix was unsuccessful, because only the resin modulus was thought to be important. Furthermore, organic solutions tried by most the materials elaborators led to a decrease in strength while increasing the modulus. Therefore, the leading idea is to increase the shear strength without affecting the resin modulus.

Continuous dialogue with organic chemist expem from SNPE during task 2 led to the conclusion that the 80 MPa resin shear yielding strength may be an intrinsic limit of polymer chemistry.

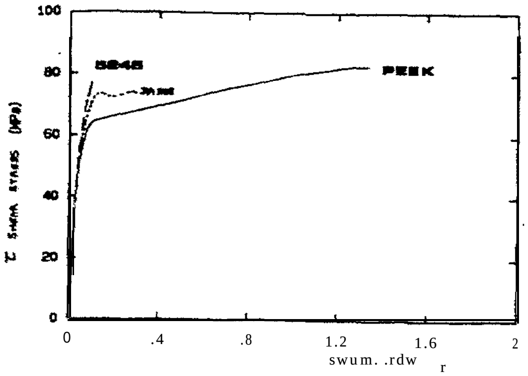


Figure 20 : Range of commeciai available resins for aeronautics. 5245 horn Narmco, DA508 (resin 1) from SNPE, PEEK from Fiberite (RAIL shear test at 20”C)

However, the ductile experimental resin number 4 has been successfully reinforced by whiskers (about 6 % in volume). The figure 19 recalls the matrices shear response and the 0° ply compressive failure strerwth.

The introduction of particles in a ductile matrix appeared to be the main idea to study in the following steps of the project.

- Reliable and economical key evaluators have been identified. Before the costly fibres prepreging operation, a simple shear characterisation of the neat resin is a fmt selection criterion with the support of the simulation tool. In order to incorporate the behaviour of the fibre/matrix interface, a tensile test on a (k 45°) laminates should confm the matrix shear improvement with respect to the requirements, in *a* real composite configuration. As the fibre/matrix load transfer ability has been shown not to be a major parameter for compression failures, with fragmentation values ranging born 32 to 60 MPa, an evaluation by fragmentation might still be interesting before the costly preimpregnation operation, to avoid major incompatibility and for tensile properties optimisation.

- Compression improvement of composites should be obtained without degradations of other properties such as :
  - Prepreging ability.
  - Laminates processability and soundness
  - Toxicity reglementat ions.
  - Environmental resistance (hot/wet properties).
  - Impact and delamination resistance.
  - Tensile strength.
  - cost.

3.3 Deve!opmemt of pilot composites (task 3)

The folIowing step of lCOMP prograrnme was to develop pilot composites with improved compressive strength compared to material 1 T (DA508 resin + T300 carbon fibres). According to the quantitative requirements estabiised during the task 2, the major effort was put on the ma&ix.

InnovCtive matrices selection (subtask 3,11

The selection of neat resin candidates for improved composite compressive strength is summarized in figure 21. The leading idea was to incorporate in a ductile resin, which should lead to an improvement of both the shear stress and the shear modulus without any decrease of the elon~ation. as demonstrated with systems 4T and ST in task ;.

- Resin 6 has been chosen to persue the work. A thermoplastic (PES) was added in resin 6 to obtain suitable rheology for industrial prepreg.

- An attempt to use a tough thermoplastic has been decided. PEEK appears to exhibit a very ductile behaviour with almost similar shear *strerygh* and modulus than top level thermoset systems (figure 20).

[Code \	Com~osition
t 6A	Experimental resin 6; PES (25 Phr) (si&ple epoxy amine) (Poly E&er Sulfone)
63	Experimental resin 6 + PES (}6.7 phi)
6C	Experimental resin 6 +PES(16.7 phr) + whiskers {18.7 phr) or 10,9 % by weight
7.1	PEEK (Polv Ether Ether Ketone)
7C.2	PEEK ~ w~iskers (100/19 wei~t)
7C.3	PEEK + whiskers (1 00/1 9 weight) with molding cycle variation compared to 7C.2

Figure 21: Neat resin candidates elaboration

Based orI the confidence of the identified neat resin shear test key evaluator, SNPE molded 6 resin plates for Iosipescu evaluations. The shear behaviors and properties are presented in figure 22.

Iosipescu shear test at room temperature

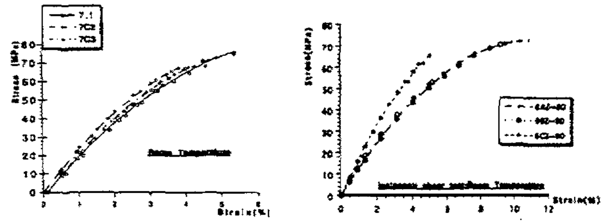


Figure 22: Task 3 neat resins shear response

For both thetmoset (resin 6) and thermoplastic (resin 7), the introduction of whiskers approached the desired resin behaviour. The best properties were obtained with experimental systems 6C and 7C.3. However, for these two materials, only the resin modulus was significantly increased. Poor quality of thermoplastic plates led to very [OW failure strain, not in accordance with Rail shear results reported in figure 20.

In the budget and time frame of the prograrnme, no optimisation of micro-reinforcements and processability were possible. **As** SNPE experts felt that the partictde routes was attractive and certainly industrially applicable with strong opt.irnisations, the consortium decided to launch the rrtanufacturing of the 2 following pilot prepregs, to generate preliminary knowledge of this concept on general relevant properties :

- T300 carbon fibre from Toray
  + resin 6C (epoxy + whiskers)
- AS4 carbon fibre horn Hercules
  + res~ 7C (PEEK+ whiskers)

Even if the selected best resins did not tldly match the task 2 requirements, the aim of these last steps was to get the compressive response of the unidi.rectionaI laminates and to evaluate for the fmt time various loading responses of laminates made out of resin + whiskers in combination with pretension.

Pilot tmoprem rnanufacturim (subtask 3 .2]

For both pilot prepregs, moving ffom the laboratory scale, where good materials were elaborated, to the production line raised a lot of problems. Beyond different processing factors, there are specially changes in the rheology characteristics by volume effects. For example, the 7C matrix weight content varied by 27 to 50% in the deiivered composite prepregs.

The relatively bad quality of laminates 6C/AS4, 7AJ'AS4 and VYAS4 has to be taken into account in results anaiasys.

Basic commission responses (subtask 3.3):

Iosipescu specimens are appropriate for fmt shear characterization of neat matrices. However a Rail shear type of test could be itttersting for better shear failures.

With whiskers addition, a reduction in failure strain with a corresponding increase in both compressive strength and modulus was observed in (k 30°)5s laminates (see figure 23).

- Addition of whiskers in a tough matrix seems to be a way to improve the shear properties of composites. Nevertheless, there is a difficulty to get their homogeneous distribution into the laminate.

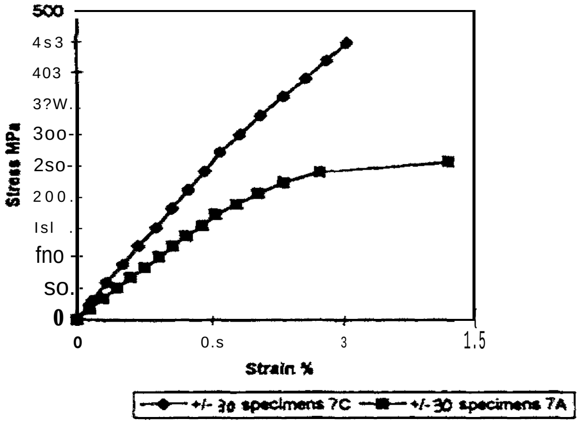


Figure 23: Influence of adding whiskers to MO°
ASWEEK kurtinate

General mechanical pmoerties (subtask 4. Q

For subtask 4.1 a thermoplastic PEEK (AS4,7C) and a therrnoset (T300/6C) matrix composite material with whisker were tested. Laminates cured with pretension and with no pretension were investigated in the case of the thermoset.

The overall aim of subtask 4.1 is the investigation of relevant properties for design in order to assess the potential of the materials solutions (i, ii) for industrial applications. The following test were performed to cover the aspects of mechanical joints, damage tolerance behaviour and environmental resistance :

- notched tensile and compression strength at different temperatures on isotropic laminates
- fracture toughness G_{IC} and G_{IIc}.

Taking into account the Iack of optimisation of materials 6C/T300 and PEEWAS4, the subtask 4.1 gave the following indications for future use of whiskers and 0° fibre pretension as composite compression improvement solutions :

- 0° fibres pretension benefit on unidirectional compression strength seems not to transpose in realistic multi-angles lay-up containing a bolt.

- The pretension operation is not industrially applicable for compiex parts. However, the use of pultruded rods, unidirectional tape instead of fabrics, and all kinds of presentations which minimizes the fibres initial misalignment, are profitable for better compression properties.

For both pilot composites, no improvement in compression on multi-angles laminates has been obtained with respect to 1 T SNPE commercial reference. However, the use of whiskers is stiI1 believed to be an attractive route to increase the matrix shear properties which govern the composite compression, If designers use common rules of stacking sequence optimisation to avoid edge effects (ply 1 by 1...), any improvement found in unidirectional strength should be transposed into real multi-angles lay-ups with bolt submitted to compression.

It is finally interesting to notice that the addition of whiskers in a ductile matrix tends to increase the composite delamination resistance and that it seems to not affect the tensiie properties.

4 ACI-UEVEMENTS AND CONCLUSIONS

4.1 Achievements summary

As the compression properties remain one of the composite design limitation, alligned with the objectives of this project, the foIlowing progress has been gained towards improved composite compression strength (1 1):

Regardin~ DheRomens understandirw and constituents r ~

- The key starting point of this cooperation has been to generate a complete data base on a variety of fibrefmatrix couples with an associated characterisation of both neat constituents and initial geometrical imperfections inside the molded laminates. A wide range of unidirectional compressive failure modes could be faced, which Ied to the construction of a simulation tooI. This work permitted to propose quantitative requirements on constituents to improve composites compression.

- For standard carbon or glass based composites, the compressive failure is clearly matrix dominated, during the microbuckling phenomenon of such low inertia fibres. The main conclusion is that the most effective resin shear behaviour requirement is to increase the resin yielding strength. Secondly, an increase of the resin modulus is also profitable. But with respect to the previous Iitterature reports, it is important to state that an improvement of composite compression can be obtained oniy if the combination of resin strength and modulus is increased. For unidirectional carbon composite compressive strength, the relative influence of the matrix shear strength is around 60 ‘/0, compared to 40 ‘A for the resin moduius.

The initial tibres misalignment is the third important parameter which governs the fibres microbuckling mechanism. Experimental 0° plies pretension during the curing operation has confined the influence on compression properties. It has also permit to extract a characteristic angle, which describes the complex in-situ fibres misalignment distribution.

The fibre/matrix interface shear load hansfer ability appeared to be a secondary parameter for composite compression. For interracial shear strengths ranging between 32 to 60 Ml% measured by fragmentation, and the 3D simulation conflation, it seems possible to state that the interface optimisation with regards to tensile properties w iIl satis& the carbom+esin compressive requirements.

Regardin~ materials and vrocess:

The ICOhlP target for composite compression improvement has been set to 300A. This represents the step which would be necessary to recover a more symetric behaviour between tension and compression, in order to take fill advantage in primary parts design of the second generation Intermediate Modulus carbon fibres. The following points show the main conclusions after this development programme:

- For highly loaded structures, the fibres market which is mainiy cost and ecology driven, tends to concentrate on very kirge production of a minimum of glass or carbon references. The Boron reinforcement is expected to disappear even though it was the only fibre solution to significantly improve the compression properties. This was due to a 106 factor of the stiffeness-inertia product between Boron and Intermediate ModuIus carbon fibres (Boron filament diameter = 0.136 mm, Modulus = 400 GPa, carbon tik.rnent diameter= 0.005 mm, Modulus= 280 Gpa). .4s there is no coming development of carbon fibres with comparable Boron inertia, the progress in composites compression is based on innovative matrices.

- h a fwst step, experimental resins have been e~aborated with the strong requirement of processabiIity with available industrial equipments. The demonstration has been performed that adding micro-particles like whiskers in a tough matrix could fidfil both objective of processability @epreging and laminates molding) and increasing the unidirectional composite compressive strength. It has also been concluded that the polymer chemistry has probably reached an upper limit, in terms of shear properties.

Taking into account this first experience with low properties resins, two pilot composites have bee manufactured using high properties organic matrices, tough enough to ensure a strength and modulus shea improvement by whiskers addition (ultra high modulus filament with small sizes compared to th carbon reinforcements interspaces in the composite). The target of 30 % gain in compression was no reached in the fkrne of this project, mainly because o the lack in optimisation of the particles and of th process parameters. This will require a tremendou effort in a totaly new research route for a preprege like SNPE. However, the particles solution appeare to be the unique idea by chemists experts to increas the organic matrix shear behaviour (similarity wit concrete). Furthermore, the frost trials made in thi project suggested that there were no blocking point from the processability and the economical aspects. Finaly the indications that the other properties lik tension and interlaminar resistance should not b affected are etwourageous. For stacking sequence that minimize edge effect and delaminatio sensitivity, any gain in unidirectional compressio should transpose in compressive strength of industria multi-angles lay-ups containing bolts or impac damage, which are typical aircraft design criteria.

The processing soIution which consists to put the 0 tlbres in pretension during the laminate ctig cycle did lead to an increase in compression. However thi is not seen as industrially applicable by th manufacturers of composites structures. Nevertheles it is an indication to better resort to materia presentations that minimize initial fibres misalignmen if high compressive properties are looked for (avoi fabrics, use of puhruded sub-elements).

Re ard” test delisation:

- A reliab~e test procedure has been developed to asses intrinsic unidirectional compressive strength. Loa introduction problems have been overcome by using (* 60,, 02, + 60J,D stacking sequence. This work ha been appreciated by the MIL-Handboo representatives, who are preparing a composit version, which will be the basis of most arnerican an European standards. The scientific community agree now that any direct testing of purely unidirectiona laminates will not give compressive data. Th developed procedure led to compressive faihire stres of about 2300 MTa for carbotiepoxy (- 2.5 ‘/0 failur strain) compared to an average of 1000 MPa currentl reported up to now in the Iitterature.

In order to describe the initial fibres misalignmen after laminates curing, a technic involving dat processing of polished cross-section has bee developed; it permits to reconstruct the real in-situ 3D fibres path and to compute a representative waviness parameter to be used in simulation.

An important conclusion of that programme is to have identified an economical key evaluator, As the composite compressive strength is matrix dominated, before launching costly prepreging operation, a shear test on neat resin appears to be a reliabie selection criterion to detect any gain in composite compression.

Combining this test information with the develor)ed 3D sirnuhtion tool, taking into account fibres waviness and non Iinear matrix behaviors, the potential compressive properties of a proposed fibre/matrix combination can be forecasted.

4.2 **Perspectives and conclusion**

For light weight composites made out of continuous carbon or glass fibres, the compressive strenglh improvement is still a very high challenge for materials elaborators, This project clearly demonstrated that the compression failure is matrix dominated, but it also suggested that thermosset or thermopkistic resins may have reached the maximum shear performance available through polymer chemistry. Therefore, the addition of micro particies is seen as the on}y solution 10 enhance the resin properties. However, the optimisation of such micro-reinforcements may require several years of research, which will be followed by around 10 years to reach qualification on an aircraft application.

For the aircraft industry, one of the main materials objective is to reduce the cost of the selection and qualification. Such cooperation was a unique opportunity to have much quicker iteration than in the past between structures designers requirements and material supplier response. In the field of composite materials, there is a need of a real effort for phenomena understanding, in order to address quantitative constituents requirements. Otherwise, large research efforts can be put on secondary properties.

As long as the dysirnetric behaviour between tension and compression will be very high, most applications will take much more. benefit from compression improvement rather than from firther fibres or even tougher resins development. It is very important to contribute to reoriente partially the research effort on better shear resistance matrices.

5- ACKNOWLEDGEMENTS

We would like to thank all colleagues that made the accomplishment of this project possible. The cooperative attitude of the partners during the programme is grate fi.dly acknowledged.
The consortium wishes to acknowledge the CEC for their support to this project.

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(10) - BRE2-CT92-0314 contract and technicai annex

(11) - ICOMP final technical report

Ilrite Em-am lCOMP~ 13 RE2-CT92-(13%4	**INNOVATIVE CONSTITUENTS FOR IMPROVING THE COMPRESSIVE STRENGTH OF COMPOSITES**

DOCUMENT REFERENCE No : PR-0003-D ISSUE :1

TITLE

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~&%iiiepo%~o CEC}DGXH (working period february 1993 to july 1994).

ICOhIP programme, proposal BE 5880.

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PARTNERS:

company	Technical representative
DAS	VAUTEY
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UP	MARQUES

* Signature of the programme coordinator: P. VAUTEY

~ 4 *

Summa-y :

According to the minutes of the meeting held in 13russeis with Dr Cervera-March (MN- 1005-D/0), the mid term report is delivered with respect to the contractual planning. This document has been finalized during the mid term assessment meeting.

The objective of the first half of the programme was to make a significant improvement in the understanding of the composites compression mine modes. Although some key experimental materials elaboration delayed the completion of the ana[ysis, the successful developments of new testing procedures, of fibres misalignment measurements, of a 3D simulation tool validated on a wide range of fibre/matrix couples, should permit to match the objective of task 1 and to specify constituents requirements to try to improve composite compressive strength in a second step.

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* Contents aid actions approved by partners when signed by (I1c coordinator ~lier PCC comments.

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Doc. DGT/DEC/hJT N“ 59.211
PR-0003-D/l
October 4th, 1994

lCOMP

BE 5S80, BRE2-CT92-0314

MID TERM REPORT TO CEC/DGXH (To +18 MONTHS)
&
THIRD PROGRESS REPORT (MONTHS 12 TO 18)

Project title : Innovative constituents for improving the compressive strength of composites

Project coordinator : Dassault Aviation (F)

Partners : SNPE (F), DB Q?), DLR @), DRA (UK), NLR (NL), OINERA (F), A&rospatial
(F), University of Porto (P).

For the period : february i st, 1993

to : jldy 3 lst, 1994

Index :

1- SCOPE AND FIELD OF APPLICATION.

This document is the mid term and the third progress report due to the CECYDGXH by the partners of project BRE’2-CT92-0314 (proposal BE 58 NJ. It has been written under the responsibility of the project coordinator with the cooperation of all partners. The approbation process through the Project Coordination Cornrnittee has been used, as presented in ref. MI$lool-wl.

This document is written in accordance with the technical annex of the contract (Dec. DGT 38.444/D). It gives, for the Last six months period, information about p~ogres performed in task 1 (see appendix A), and summarizes the relevant results obtained during the first half of the programrne. A mid term assessment is proposed, referring to the technical annex objectives.

2- APPLICABLE AND REFERENCE DOCUMENTS.

MN-1 001-IX1 : Minutes of the kick off meeting.

Dec. DGT 38.444/D : Technical annex of the contract.

FE 1101-1% - Preliminary programrne for compressive experiment choice.

FE-1 102-D/2 - Mechanical tests programrnne of subtask 1.1.

PE-120 i -FU3 - Programrne of damage assessment during composite compression (subtask 1.2J

PE-130 1-0/3 - Programme of constituents characterisation (subtask 1.3).

PE-1 401-N/2 - Programme of compression tests on flat laminates (subtask 1.4).

P13- 1501-D/O - Programme for compression modellisation (subtask 1.5).

MN- 1002-D/O - Minutes of the second PCC meeting.

MN- 1003-D}O - Minutes of the subtask 1.\ working group meeting (Farnborough, 3.11 .93J.

FAN- 1004-D/O - Minutes of the third PCC meeting.

MN- 1005-D/O - Minutes of the meeting with Dr Cervera-h4arch (CEC, februar~ 11th. 1994).

PR-00() 1 -D/O - First progress report {’~() + 6 monlhs~.

l’R-0002-1>/O° - i;irsl :mnu:il rcpt~rt (’i’~) + i 2 mi)IItlm).

hTT.~ o~l .s/~ . SNpE ~id term deliverable : experimental materials elaboration.

MN- 1006-IXO - Minutes of the fourth PCC meeting.

NT-1 001-EIO - Daimler Benz contribution to subtasks 1.1, 1.2, 1.3.

NT- 1101 -NIO - NLR contribution to subtask 1.1.

3- OBJECTIVES AND STRATEGIC ASPECTS.

3.1 - recall of the mozramme main objectives.

In spite of a constant increase in the use of carbon fibre-epoxy matri composites in advanced structures over the last 15 years, their low compressive properties i one of the main mechanical limitations for a more widespread integration of composites i vital structures of any industrial field.

The recent development of Intermediate Modulus carbon fibres led to ver high specific tensile strength of composites, with a gain of 50 % over the first generation High Strength fibres. Unfortunately, compressive properties, which are mainly governed b the matrix, did not improve at all.

Most of the complex structures are subjected to alternate tension an compression loads at an equivalent stress level (robots arms, boats mats and keel, aircra fins.. .). Because of the disyrnetric behaviotir between tension and compression, it is n possib~e to take advantage of the very high properties in tension, as the compressive loadin becomes the design limitation (see table 1).

	0“ tmidirectionai tensile strength (MPa)	0° unidirectional compressive strength (MPa)	Approximate year of availability
1+s [High Strength carbon fibre)	1700	1600	I 970
IM (Intermediate modulus carbon fibre)	2600	1600	1985

Table 1: Evoiution of the composites tensile/conlpressive strength balance (datas for 60 volume fibre content and a typical 180”C cure semi-toughened epoxy).

The aim of this project is to develop pilot composites having compressiv strength 3 0°/0 higher than all the current carbon-thermosetting matrix composite Manufacturing technologies should allow to process these new composites without maj changes in existing facilities.

3.2- Proiect ammach.

The appendix A summarizes the work planning of the programme as se~ u by partners and agreed by CIW'DGXH.

Task 1:

Development of a simulation tool in order to quantifi the relati,'e effect the many material parameters that influence the composites compressive strength.

Only a numerical computation seems to be able to reproduce a realisti fibres arrangement in the laminates and to deal with elasto-plastic matrix behaviour.

In order to vaIidate the modei on a wide variety of compressive failur scenarios, the key point is to generate an experimental date base which systematically gives :

- Constituents mechanical behaviors.
- Initial geometrical impetiections within the bminates (fibres misalignment).
- Composites compressive response.

Several composites families will be studied, through various reinforceme types and through proto~pe resins with different shear behaviors to find out the governin parameters.

Task 2:

Specifications of constituents properties to improve composite compressiv strength.

Task 3:

Development of pilol prepregs from specifications and innovativ constituents to increase compressive properties.

Task 4:

Characterisation of general properties and evaluation of industrializati developments before applications.

- TECHNICAL ASSESSMENT.

The mid term of the project arrives six months prior to the completion of task 1, ~,hi~h is not in paraiIel with any other work package (see appendix A).

In order to discuss the technical progress, it is recall hereafter that task 1 will mainly result in :

Simulation tool for compression description from constituents properties.
Prototype resin systems for better key matrix role understanding.
Innovative testing procedure to characterize unidirectional composite compressive behaviour.
~l$_{ew}$, t$_{ec}$~i$_{c}$s to identi~ initial fibres arrangements within the 0° plies.
Associated constituents characterisations.
Composite tests and damage investigations during compression.

4.1- Surnmarv of the suecific objectives for the relevant period.

Selection of fibres and matrices to support subtask 1. i experimental investigation.
Writing of subtask 1.1 detailed test programme to improve significantly the understandin of the compressive failure modes through a variety of fibre/matrix combinations.
Choice of the ‘‘Unidirectional compression test”.
Harmonisation of damage assessment technics (subtask 1.2). ~
Writing of subtask 1.3 detailed test programme for constituents characterisation.
Prepregs delivery and laminates moulding.
Writing of subtask 1.4 detaiIed test prograinme, and beginning of mechanical test on th reference material.
Writing of subtask 1.5 detaiied programme, beginning of parametrical computation for ke constituents characteristics identification.
Specimens manufacturing.
Completion of the mechanical tests on the reference system (subtasks 1.1 & 1.4).
Constituents characterisations (subtask 1.3).
Beginning of mechanica! evaluation on experimen[aI composites.
Cornpietion of the simulation tool development.
Assessment of damage growth during compression on laminates containing a hole.
Experimental demonstration of initial fibres misalignment influence on compressiv strength.

4.2- Critical overview of progress.

4.2.1- **Subtask L1.**

A detailed test programme (PE-1102-DII) has been approved by the working group partners and the Project Coordination Committee.

Basic fibres have been selected taking into account the materiai supplier (SNPE) requirements for prepiegin~. The various fibre~matrix couples are presented in appendix B. They should provide a wide range of compression fai~ure modes, which should permit to identify the governing parameters.

Materials delivery:

Prepregs with the reference resin have been delivered, and laminates have been moulded and C-SC~" by Daimier Benz (see appendix D).

Prepregs with the 5 experimental resins have been delivered. That part being very important for future resin optimisation, SNPE has needed more time than scheduled to formulate 5 new resins and to prepreg various reinforcements (see in appendix G a summary of the deliverable report NT-1001-S/0 from STJPE about ali materials elaboration).

Dassault Aviation has moulded all the plates made out of the experimental formulations. The appendix C gives an overview of the materials delivery. Major difficulties have been encountered with experimental prepregs (4T, 5T, 4V, 5V), because of very short shelf life, of the poor tack and of the very limited resin flow during process (see appendix E). For mechanical results interpretations, it will be important to bear in mind the laminates fibres content and the associated ply thicknesses.

Mechanical tests progress :

All the test procedures have been selected. It has to be noticed that a significant improvement has been realized in the unidirectional characterisation in compression : the use of a special stacking sequence [+607 .0~,t603)s in a modified ceianese loading device led to reliable and satisfactory values.

For the reference material (T3001DA508), 0° ply strain to failure of 2,5% have been measured, compared to - 1,5Y0 currently reported in the litterature fo such a carbordepoxy. The first results obtained by DLR, who is in charge of all the 0 compression tests, are satisfactory. The successfi-di transfer of this evaluation method t DLR demonstrated the good reliability of the procedure.

The detailed progress of mechanical tests and available results ar presented in appendix F, referring directly to the tests matrix as established in subtas 1.1 program (PE- 11 02-D/1). .4bout 8(Y?Ao of the tests are performed so fm. Due t processing problems, specimens made out of experimental resins became available jus recently.

4.2.2 - Subtask 1.2.

The work carried out in subtask 1.2 is in direct support of subtasks 1. and 1.4. The objective of this subtask is to study the sequence of damage events th occur during compression loading of unidirectional and multidirectional laminates an to determine the mode of compression failure by fractographic analysis. Additional tes will investigate the compression failure modes of the individual constituents i.e. th fibre and the matrix. These fi-acture surfaces will be compared with those obtained fro the compression tests on the laminates. Full details of the experimental program a given in document PE-1201-W3.

Results obtained to date as a direct support of mechanical tests analysis :

Unidirectional [on~itudinal commission : Tests campaign t determine the unidirectional compression strength on all material systems has begu recently. Once these tests are complete, examination of the fracture surfaces will begi The data obtained from these initial tests will also be used to define the loads required establish the sequence of damage event prior to failure. The results from these tests il,;i be available shortly.

Unidirectional transverse comtx-ession : The plates and correspondi specimens have been manufactured in accordance with PE- 1201-R/2. Prior compression loading, non destructive tests for quality insurance were performed uhi found that the material 1 Y contained microcracks after manufacture. These(cracks a thought to be the consequence of the thermal stresses induced during curing and the lo interfacia] properties of high modulus fibres. Mechanical testing on all material s;ste is comple(ed as we] 1 as fracture expertise.

+45 ° compression tests : Room temperature tests on all materials have been performed. ;uring loading, photographs were taken of the prepared specimen edges. The results obtained are being evaluated at present.

345° tensile tests : A detailed damage inspection of a specimen loaded al approximately 95% of the static strength has been performed on the reference material 1 T. Microcracking was detected by C-SCAN with the main defects being intralaminar cracking. Futher tensile tests on other systems will be performed and a damage inspection will be carried out if the stress/strain behaviour shows any abnormalities. The ultimate aim of this part of the project is to check whether the stress yield value, which will later be used for modelling (subtask 1.5), corresponds to the same failure mechanism for each material.

Fibre failure : Loop tests to measure the compression failure strain of individual fibres have been completed. Work is about to begin which will study the fracture surfaces obtained using a scanning electron microscope (SEM). This will permit to check the ftilure mode (between bending and compression) for results comparison, and to give indications of the various fibre microstructures response in this loading configuration. The SEM fracture investigation after compression of single filament embedded in matrix will also help to identi~ the mine mode of laminates subjected to compression, by SEM comparison.

Other tasks, development of techniques :

Fibres misaliwu-nent measurement : The development of a new technique has been completed (see description in appendix H]. It mainly covers 2 objectives : i) .As a support to the experimental pre-tension work of fibres during curing, statistical misalignment have been measured to provide the first correlation with the unidirectional compressive strength. ii) The fibres misalignment evaluation for each type of materials, which is currently in progress according to plates sample delivery, will be a key input for the simulation tool (description of the fibres waviness).

Acoustic emission : h order to monitor damage events during loading, an attempt to use acoustic emission is made. Due @ the small amount of available material and to the fact that compression specimens must be short to avoid buckling, a wave guide technique has been developped to measure acoustic events on these small coupons. Although it is less sensitive than a direct contact, noticable differences between the various fibre/matrix couple seemed to be observable. The technique is now ready to be applied to other materials during 0° compression.

]n-silu ultrasonic inspection : The [echnique seems to work very]vel 1. ‘I-lm datas acquisition is going on. The analysis of ultrasonic signature ailer each loading !cvcI is on proytss,

4.2.3- Subtask 1.3.

All the tables and figures which are illustrating the followin statement of subtask 1.3 progress are presented in appendix I.

Materials delivery :

All the fibres (Carboli T300, M40 and h160 - Boron from AVCO, E gkxss from VETROTEX} have been distributed by SNPE.

Laminated plates made out of the reference resin DA508 have bee delivered. The neat resin p~ate and the prepreg with h440 fibres had been produced agai by SNPE, following transpotiation and moulding problems.

Experimental resins RI to R5 have been formulated by SNPE. Th main difficulty w% to match resins stresskrain behaviors, which were requested b mechanicians to identi~ matrix governing parameters on composites compression, i addition to prepreging feasibility requirements. This key task took more time tha scheduled.

All systems are epoxy-based with an hardener :

- RI also contains some CTBA{.
- R3 is a very ductile resin with a great amount of CTBN.
- R4 is the same as R3 plus 6$6 (Vf) of whiskers.

Resins plates were moulded on February 1994 and delivered on Marc 1994.

Fibres characterization :

<u>Compression of elmbedded filaments</u> (ONERA) :

Experiments with T300, h440 and boron fibres were satisfi l achieved. Some problems arose for the other fibres :

- For M60 fibres, the fibre is already broken after the specimen curing
- For glass fibres, the specimens sometimes buckle before the first failure ar results are scattered.

Results of strains at first failure (uncorrected and corrected c residual thermal strains) are presented in Table 1. In Table 2 are reporte corresponding values for the saturation state. Results reported in Table 1 are ak reported on Fig. 1.

Loop tests + fractography (DRA) :

The loop tests to measure the compression failure strain of th fibre, have been completed. Due to the time required to complete each test, 1 tests were conducted on each fibre and the results are listed in Table 3 and als shown graphically on Fig; 2.

Transverse compression tests @RA) :

Test will start after all the pull-out tests have been completed.

Matrice characterisation :

Matrices tensile behaviour - Systematic characterisation (SNPE) :

Tensile properties are reported in Table 4 and the correspondin curves are reported on Fig. 3.

Matrices shear stressktrain behaviour (ONERA) :

Tests were preformed on “Iosipescu” specimens which a represented on Fig. 4. A scheme of the testing device is reported on Fig. 5. A strength results correspond, in fact, to tensile failure initiated from notches, th only relevent results from the tests are the shear beha~’iours Up to this ultima level.

Results corresponding to initial tangent modulii are reported Table 5 and also presented on Fig. 6 vs the tensile modulii as determined SNI’E.

~ (D B) :

The specimens were manufactured in accordance to Dec. PE-1 301-0/3. One strain gage (HBM : 6/120 LY 11) was bonded (adhesive : HBMZ70) on each specimen.

Preliminary tests were performed and demonstrate that it was impossible to reach failure of neat resins under compression. Therefore, it has been decided to switch to a toughness characterisation of matrix with a edge notch tensile test.

Fibrehnatrix interfaces :

<u>Fibre/matrix load transfer</u> (ONERA) :

Prelixnimuy tests shown that the fragmentation tests cannot be carried out with boron fibres for which the first failure induces the total failure o the specimen. For glass fibres the saturation state, necessary to induce the critical length and then the transfer shear stress, coutd not be reached before the failure o the specimen.

For the other fibres (T300, M40), the results of the tensile tests carried out far assessment of strength statistic are presented on Fig. 7 and 8. Tests were performed with 4 different gauge lengths. The Weibull parameter "m" can then be obtained and used to compute the tensile strength at critical length.

The fragmentation tests programme began with adj ustement o experimental parameters for coating the Flbres with DA508 resin. As the failure strain of DA 508 in not significantly different from the fibers ones, the saturation at the end of the fragmentation cannot be attained. This leads to a coaxial geometry where the fibre is coated with matrix under study before being embedded in a tough epoxy resin **(DGEBA LS556** with 27 % **HT 972}. The** main difficulty is to realize a resin sheath of uniform thickness without any droplets due to the Rayieigh instability.

The results obtained with 3 types of fibres coated with DA5(IS :

- treated and sized T300 {T300)
- untreated and unsized T3t30 (UT300)
- ~reatcd and sized M40

are presented in Table 6 and Fig. 9 where :

$T_m = \frac{\text{CT, (Lc).r}}{t\text{-}_c}$ is the interracial shear stress with :

L_C = ~ L. is the critical length (L. is the mean fragment length)

~, [L_C) is the tensile strength at the critical length

r is the fibre radius.

Characterization of interfaciai capability of other fibres (R1 t R5) was carried out with T300-5013 fibres. As matrices physical characteristic were unknown, coating of the fibres was firstly done with the same conditions a for DA508. As the resin film seemed too thin two other coating conditions wer experienced (resin bath temperature, velocity of the moving resin bath). Th results are reported in Table 7 and aIso on Fig. 10.

<u>Pull-out tests</u> (IX-A):

Tests on the lT and IT’ systems (T30MDA508 and untreate T300~A508 respec~ively) are completed and the variations of Interracial She Strength (1SS) against embedded lengths are reported on Fig. 11 and 12. On th diagrams the points are the experimental data and the continuous lines are th trend calculated using the experimentally determined maximum 1SS. Th maximum 1SS for 1 T and 1 T’ systems are 157 MPa and 115 MPa respectively ar show that the 1SS is significantly increased by surface treating of the fibre.

Work is currently ongoing to measure the maximum 1SS of tl 2T and 6T systems (experimental resins R1 and R5 respectively).

4.2.4- **Subtask** 1.4.

The aim of this package is to study how unidirectional experiment composite properties translate in multidirectional iaminaies. A de~ailed test programn was prepared by the NLR, the working group leader of subtask 1.4. This document (P 1401 -Nf3 May 1994) describes (un)notched compression tests, loughness tests and study of the effect of pre-tension of 0° plies on compression strength. One base materi DA50WHO0 and two experimental materials (2 and $) \\i 11 ix considered. ‘l- appendix J recalls the tests campaign.

Materials delivery :

All IT laminates were moulded and distributed to partners by DB. AS also received some 1 T prepreg from SNPE for the pre-tension experiment.

The experimental materials 2T and 6T have been selected respectivel for S and L Problems have been encountered by SNPE to apply the prepregin operation to a much larger material quantity on an industrial line. Extra resin rheolog and behaviour investigation were made. From this, the prepreg 2T could be elaborate and sent to Aerospatiale on September 1994. For material 6T work is still going on. I no sucess before mid October, 6T laminates will not be mechanically tested withi subtask 1.4 (see MN- 1006-DIO for details).

Tests progress :

The first trial of the pre-tension of 0° plies during the curing proces was not successful, based on a compressive test evaluation (see the developed devic in appendix K). A second trial is going on. The 0° clamped layers were pre-cured i order to avoid any slip during the curing cycle, when the resin goes through a minimu viscosity state. In all cases, laminates are sent to IMFL for fibre waviness measurement.

The appendix L summarizes the progress of subtask 1.4 and gives th available results. The late delivery of 2T and 6T materials will delay the multi angl laminates mechanical evaluations,

4.2.5- Subtask 1.5.

Dassauh-Aviation issued a document (i?E- 1501 -DIO) that describes th axis of development and the work sharing between involved partners.

The starting point is to consider that composite compressive failure i due to fibres micro-buckling, which is mainly governed by fibres initial misalignmen and the matrix stress/strain behaviour.

A finite elements mesh which represents an elementary cell of fib and surrounding fibres + matrix has been developed by D-Maul r (see appendix M) Parametric studies have been completed to define the 311 cell dmt will be used as th optimisation tool in task 2.

The University of Porto has successfully developed his own simulation model (see MN- 1006-IMI). As one of the most difficulties is to identiFj the describing parameters of materials behaviors to be used for computation inputs, it has been decided to have first a seperate anaiysis before a exchange of point of view between Dassault and Porto.

With the support mechnical tests of subtask 1.4, the University of Porto will calibrate an industrial criterion such as a “point stress” to compute stress concentration and damage diffision in the case of compression on multi-angles laminates with a hole.

Aerospatiale effort has been transfer to subtask 1.4, in order to try to experimental demonstrate the key effect of fibres misalignment, and to suggest a route for improvement.

4.3- Objectives for the next ~eriod.

4.3.1- For completion of task 1 (from august 1994 to january 1995).

Prepreging 2T and moulding plates of 2T and 6T materials for subtask 1.4.

Mechanical tests on experimental materials (subtasks 1-1, 1.4) will be completed. The remaining work volume is about one third of the overall tests campaign.

Damage characterisation under compression loading (subtask 1.2).

Point stress criterion calibration for multi angles laminates with ho[e under compression.

Ail the fibre misalignment distributions will be identified to support the pre-tension work and the simulation tool validation.

OveralI data analysis. See appendix IN and IvIN-l 006-D/O which defines the consortium strategy and pianning for all the experimental results synthesis.

Validation of the model with the experimental data base on a wide variety of fibrehatrix couples. Establishment of’ relationships for key parameters use in the cell [o reach a reliable simulation tool.

Deliverable reports for al! subtasks 1.

Beginning of the constituents specifications with the numerical tool. Preliminary discussion of the routes of composite compressive strength improvement with the materia elaborator.

4.3.2- Recall of the folIowing steps of the project (see appendix A).

Task 2:

Specifications of constituents properties to improve composite compressiv strength.

Task3 :

Development of pilot prepregs from specifications and @novativ constituents to increase compressive properties.

Task 4:

Characterisation of general properties and evaluation of industrializatio developments before applications.

5- EXPLOITATION PLAN.

Except a delay caused by prototype materials elaboration, the consortium is confident to matc the objectives of task 1, which will @d to constituents specifications. A “GO TO CONTINUE decision was approved and wished by ail participants during the iast project meeting (see MN 1006-IXO). The resources initially scheduled for the foHowing phases remain valid.

No patent has been taken up to now. The material supplier mentioned that resin formulations ar usually not patented. However, neither resin formulations nor fillers use should be release outside of the consortium.

It appears to be too early to state about market potential, as it is too soon to see if the target c 3 O*A composites compressive strength increase is achievable. As already mentioned, if th balance between tension and compression is reached, this will have a tremendous impact o composites applications. However, before quantitative constituents requirements are identifie the overail target of organic composites compression improvement is still seen as a risky proje (see CEC inquiry form, included in MN-1006-D/0).

6- MANAGEMENT AND COORDINATION ASPECTS.

- The performance of the consortium lies in the fact that partners are compIemeritary. Indee vehicules manufacturers @assault, A&rospatiale, Daimler Benz) mainly speci~ som requirements, research institutes (DRA, ONERA, DLR, N~LR, I_J.of Porto) bring their experienc about materials physico-chemicai knowledge and mechanical testing, while a continuou dialogue is engaged with the composite elaborator (SNPE).

  As the validation of an innovative simulation tool is based on a large number of experimentatio to study several compressive ruine modes associated to various composite fmilies, the followin organization procedures are used :

  - Rigorous management of more than 1000 specimens.

  - Exchange of ***row*** materials, lamiria[es, coupons ***using*** dispatching note and a uniqu codification for the entire project.

  - A common strategy has been adopted for resuhs presentation in order to facilitate a ric analysis of the data base by all partners.

  - First drafts of task 1 deliverables with available results will be exchanged between partne 3 months before contractual completion, in order to have enough time for resuIts analysi and simulation tool validation.

  - Each of the five subtasks 1 have been shared between partners, in terms of coordination. synthesis of the managing organization is given in MN-100 1 -Dfl. The appendix N give the deliverables presentation for task I (To + 24 months), that have been adopted by th project steering committee (see IMN-1006-IYO).

  From the communication point of view, four types of exchange have been engaged up to now :

  - Overall programme strategy and testing progress presented in march 1994 at th “International Symposium on Advanced hfiateriais for Lightweight Structures’ 94 Noordwijk.

  - Involvement of european research institutes, working on compression prior to the prese project through the GARTEUR organisation (Group for Aerospace Research an Technology in EURope).

  - Exchange with FAA (Federal Aeronautical Agency) about compression testing procedure Standards interest for the reliable test developed in the consortium.

  * Composite compression model ; to bc published by lhe University of Porto.

- The man month consumption is, up to now, in accordance with the one scheduled in the technica annex. Only minor transfer of efforts within task 1 have been done (see MN-1002-D/0) to matc with the detailed programmed work sharing.

The forr-mdation of prototype resins, matching imposed stresslstrain behaviour for a bette understanding of matrix governing parameters on composite compression, appeared to be ver very difficult and time consmnrning. This experimental data generation being very important fo the simulation tool calibration, a delay of 6 months, which has been approved by CEUDGX1 (see IMiV-1 CJ05-DW), appeared to be necessary to compiete the objectives of the programrne firs phase (task I). The appendix A presents the planning arrangements which should permit t continue the programme with minimum overall delay.

7- CONCLUSION: wurk~row-amme review.

Referring directly to the mid term assessment review criteria of the technica annex (dot 38444/D august 27~, 1992), the following statement of progress can be made :

- Although some key experimental materials elaboration delayed the completion of the first half the program (task 1 from month O to 18), a significative improvement in the understanding composite compressive ftilures should be reached through :
    - The development of reiiable. composite compression testing procedures as weH a constituents or imperfections characterisation technics (compression properties individual fibres, 3D pattern of fibres misalignment, damage growth under compressiv loading...).
    - The constitution of an homogeneous data base on a wide variety of fibre/matrix couple This is the first and unique opportunity via the consortium to study compressive failur scenarios of different composite families which are used by several industrial secto according to the reinforcement nature. The 6 months delay of task 1 comes from prototyp materials and resins elaboratioils with very severe mechanical specifications. This stud was a key activity to be able to distinguish the governing parameters of the resin whic mainly pilotes the compression ruines.
- The riched data base obtained with reliable compressive tests should lead with confidence to Ih development of a 3D simulation tool.

Future exploitation strategy remains unchanged ivith respect to the initial targe and planning (see appendix A). All partners really wish to eon{inue, because after the completion task 1, the consortium should possess a clear understanding and a powerful simulation tool specify requirements on constituents (fibre, matrix, interface) to improve composite compressi strength.

The following step will take advantage of the consortium, which involv materials users and supplier, in order to propose innovative and feasible solutions of improveme The prototype composites will then be evaluated with the help of the other laboratories.

Doc. DGT/DEC/MT N° 59.211
PR-0003-D/1
October 4th, 1994

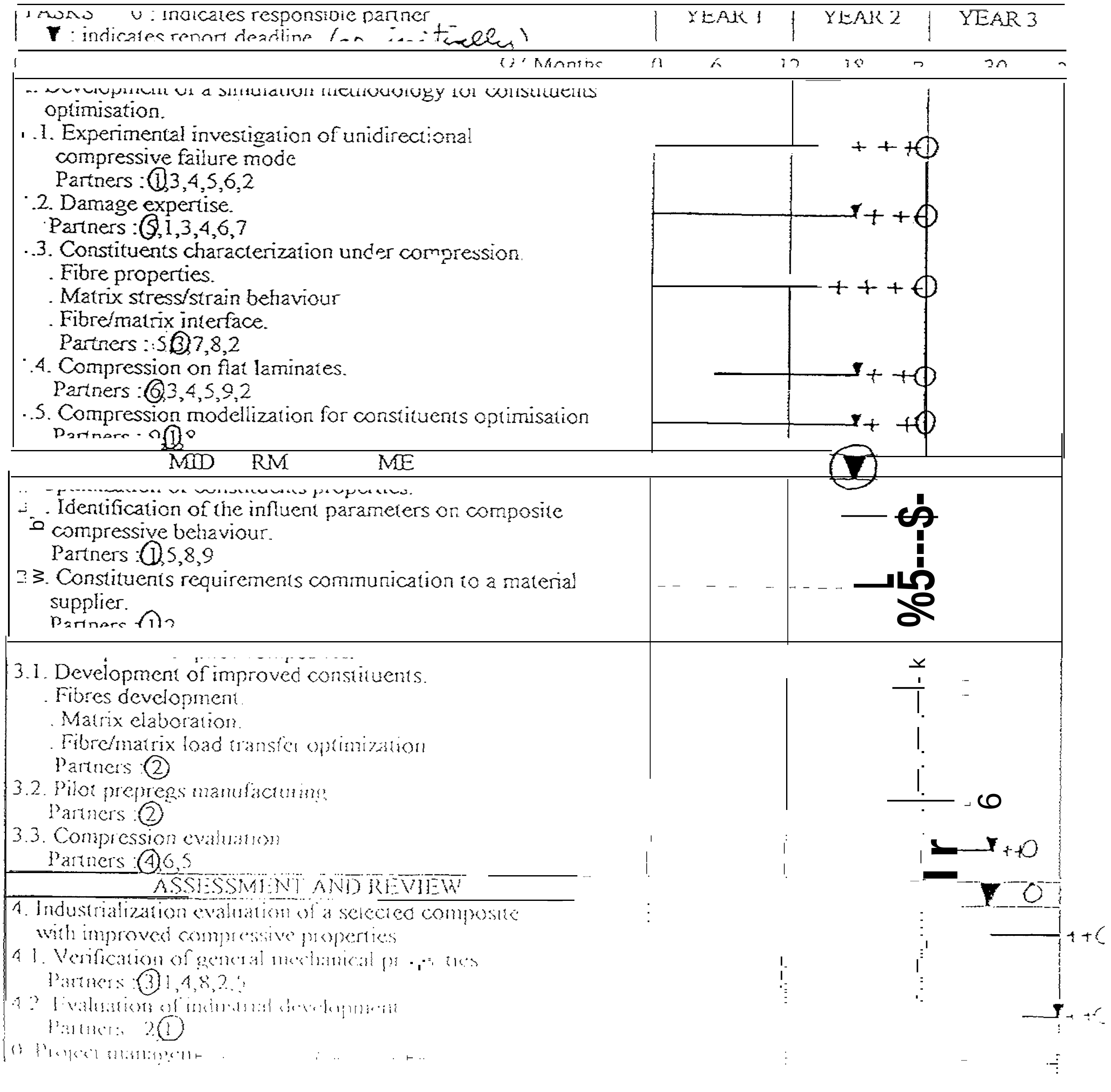
APPENDIX A

WORK PLANNING CHART OF THE WHOLE PROGRAMME

(revised deliverables planning according to MN-1005-D/0)

○ : new deliverables
+++ : delay for experimental systems analysis

remark : results on the reference system in task 1 should be available at To + 18 months for the mid term assessment.



TASKS ⓪ : indicates responsible partner

optimisation.

.1. Experimental investigation of unidirectional compressive failure mode
Partners :①3,4,5,6,2

.2. Damage expertise.
Partners :⑤1,3,4,6,7

.3. Constituents characterization under compression.
. Fibre properties.
. Matrix stress/strain behaviour
. Fibre/matrix interface.
Partners :5③7,8,2

.4. Compression on flat laminates.
Partners :⑥3,4,5,9,2

.5. Compression modellization for constituents optimisation

. Identification of the influent parameters on composite compressive behaviour.
Partners :①5,8,9

. Constituents requirements communication to a material supplier.

3.1. Development of improved constituents.
. Fibres development.
. Matrix elaboration.
. Fibre/matrix load transfer optimization
Partners :②

3.2. Pilot prepregs manufacturing
Partners :②

3.3. Compression evaluation
Partners :④6,5

ASSESSMENT AND REVIEW

4. Industrialization evaluation of a selected composite with improved compressive properties

4.1. Verification of general mechanical properties
Partners :③1,4,8,2,5

4.2. Evaluation of industrial development
Partners : 2①

0. Project management

APPENDIX B

FIBRJHMATRIx COUPLES TESTED IN SUBTASK 1.1 TO STUDY VARIOUS SCENARIO OF COMPRESSION FAILURES

	Fiber / Matrix	~ R3mfl frorll A VCO Jx;	**m'fca l-bon** M4G from TO RAY x	*HMc~rLmn* N160 from TO R/W Y	**I%Rca** rbofl T300 from SO FICA R T	EG12ss from VETI.70TEX V	T3Uu poor interface P
1	RefereRcc matrix DA 508 s~p~	c 20"C 9I " & F 12CPC	c R'I- 1 1 F	; F	C } HT M } I' } F	C } HT I } F	C } 20°C I' } & 120°C
2 3 f 4 5 6	5 cxperin?el J1al ! I'eSi Jzs wiii] different G/S I“2tios {SNPE)				C } M } 20°C & 120°C I' }	C	
I	Ist variation of fibre vo~ume conte~lt (about 50 %)				(= c	l	=
1	2nd variation of fibre voIume]contat (xbout 65 %)				C (1T“)		!.

X5

E+T : Tenpmmre effecl if resin Tg allows il :

20”C no aging, reference case
70°C no agin~
120°C no aging
140”C mo aging

F : Fibrc characterisation to k pafiormed in subtask 1.3.
i or 1': interface charackrisalion to be performed in sub(ask I.3.
M : h4atrix clmrac{crisation [o be performed in subw.sk 1.3,

C : Cornposik.s chamckrkaticm LO be performed in suksk i. 1

detailed evaluations which stand under C for each fibre/matrix couple

. $(\pm 60_2, 0_2, \pm 60_3)_S$ compression (5 specimens per case $\uparrow_{\rightarrow}$, $\uparrow$)

. $(\pm 60)_{10}$ compression (3 specimens per case $\uparrow$. $\uparrow$).

. $(\pm 45_4, 90_{10}, \pm 45_4)$ compression (3 specimens per case $\uparrow_{\rightarrow}$, $\uparrow$).

. $(\pm 45°)_{10}$ compression (3 specimens per case $\uparrow_{\rightarrow}$. $\uparrow$).

$(\pm 45°)_S$ tension (3 specimens per case $\uparrow$ . .).

[illegible]

E@ ‘7’CT92*314

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october 4th, 1994

APPENDIX c

PROGRESS OF MATERIALS DELIVERY

[MOuLDING PM.TES pffoGRESS

BE 5S%0 ICOMP, SUBTASKS 1,1 AND 1.4

DELIvERED MATERIALS {COMP TO PARTNERS, *September, 1994*

i5UBTASK ;, I 1;

MATERIAL] PARTNER	ıx [	IY[l W]	fp	IT'	IT.	IT12Tj3T14T15Tl	6Tjl	V12'<13V	[4V	/5V]	6V]	
@cl	x	X i X	x	x,]	x'	“...x:...:~:: x:. +~,'?~' :,::X	i 'w:	: %	, ::y	.x	,x. #	, .x. J
s	x	x –	x	x	x .	'.. x;.:. :.:.x. / ,: X.;;.: }X:21.. J	J	:,f;	.;;~ '	~	x J #	“'.x.:!
@E	x	x ; ; '	x	x	x	~ .+ .x / .x.::J~, .\ .x	x: '	, {x:	“x '	x	x j #	x
L	- X	- y ~ ~ l--y-”		x	x,	X..j : ,x .[, x:..] :...:~[.1 x	.x,”,.	, ., x :.;	..,;~	x	x #	x l
R	x	X i x (x		x	. . x.	. ~.:.j;:..x ~~ } :;~; .;.$~;.: . ~	y.;::,+	c:..,	.:x	~	x ~ #	X j
N	x	X l x [x] x {				x. ,:{.:x: j ; ,x;:/ ;.:,,x:/...x	X.J	::~::	.,;;-	x .	..X. ~ #	x:]

TEST
TENSICIN +1-45c
ILS
COMP 90”
COMP 0°
COMP 60°
caJP +/-45”

#= no plates [the prepreg is vefy dry)

SUBTASK 1.4	MATERIAL	1T	2T	6T
@A	(Pretension)	X	0	
N		X		
S		X		
@E		0		
L				
O		X		
U		X		
R		X		

(@E = Laminaics manu!acture~

X = DELIv~CY OF PLATES: O = **DEL/ LI’ERYOF F’REPREGS**

P +STRETCH M
QMP In **SEM + XI**
OM
MP
ITIAL FfD WEV,,
COMP+TE SION
CV .2C
Es?

Recall: For each exchange of materials, don’[forge? !o send a dispa:c+rjg nole to the co~-~ina~or

APPENDIX D

LAMINATES MOULDING WITH THE REFERENCE RESIN SYSTEM

(DB **comments, subtask 1.1)**

Ali laminates given in Doe. PE-I 102-D/~ were manufactured and delivered to partners.
The resin and flbrG content of ail prqxeg materials were **determined in accordance to AITM test procedufa, The resulk are given in table 1.**

Prepreg	Fibre content [%]	Resin content p4]
DA508/130re (1 W)	**68,8**	**37,2**
i.IA508/M40 (1x}	**67,1**	**32,9**
DA508/M60 (1 Y)	**63,2**	**36,8**
13A508fi300 (IT	**63,2**	**36,8**
DA508/T300 44% (1 T’)	**6?,6**	**38,4**
DA!5U8f1300NT (1P) ~	**66,7**	**33,3**
OA508/Verre (ıV) ‘	* 67,4	32,6

Table 1: Fibre **and resin conten? of the prepregsysterns**

The DSC curves of al{ p(epregs were determined in accodance to AITM test *pfocedwe,*
The following comments concerning fhe perpreg quality shoki,be made (ah given in the t?B report dated 4fth Nov,93):

DA 508/M60 (<Y)

- **f~uff of fibre on the prepreg**

DA 5081’T300 NT (f P)

- **voids between fibres**
- **unsteady** fibre alignment
- **wavy prepreg**
- **irregular pfepreg Gdge** (foil do~s **not correspond with the fxepreg)**

DA 508/Bore (1W)

- voids between fibres

APPENDIX E

LAMINATES MOULDING WITH THE EXPERIMENTAL RESINS SYSTEMS ASSOCIATED TO GLASS AND CARBON REINFORCEMENTS

(DAS comments, **subtask 1.1)**

COMMENTS ABOUT LAMINATES MOULDING WITHIN SUBTASK 1.1

(experimental resins 2-6 with carbon fibres T

material reference	prepreg tack	pre-compaction under vacuum	autoclave curing cycle	remarks
2T	OK	every 2 plies at RT	1h at 140°C 1h at 180°C under 7 bar pressure +vacuum 250/600 mbar	thickness OK: FV = 0.632
3T	almost none	ply by ply at 30°C	1h at 140°C 1h at 180°C under 7 bar pressure +vacuum 250 mbar	prepreg cutting very difficult thickness OK: FV = 0.588
4T	very low	ply by ply at RT	1h30 at 140°C 2h30 at 180°C under 10 bar pressure +vacuum 350/450 mbar	need of adhesive tape to keep in place the stacking sequence no resin flow observed after curing thickness problem: FV = 0.466
5T	almost none	ply by ply at RT	1h30 at 140°C 2h30 at 180°C under 10 bar pressure +vacuum 350/450 mbar	need of adhesive tape to keep in place the stacking sequence no resin flow observed after curing thickness problem: FV = 0.494
6T	almost none	ply by ply at RT	1h at 140°C 3h at 180°C under 7 bar pressure +vacuum 240 mbar	thickness OK: FV = 0.613

Lz?!4 “,m2+314

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APPENDIX E

COMMENTS ABOUT LAMINATES MOULDING WITHIN SUBTASK 1.1

(experimental resins 2-6 with glass fibres V)

material reference	prepreg tack	pre-compaction under vacuum	autoclave curing cycle	remarks
2V	OK	every 2 plies at RT	1h at 140°C 3h at 180°C under 7 bar pressure +vacuum 250 mbar	thickness OK: FV = 0.57
3V	almost none	ply by ply at RT	1h at 140°C 3h at 180°C under 7 bar pressure +vacuum 300 mbar	need of adhesive tape to keep in place the stacking sequence thickness almost OK: FV = 0.653
4V	low	ply by ply at RT	1h30 at 140°C 2h30 at 180°C under 10 bar pressure +vacuum 400 mbar	need of adhesive tape to keep in place the stacking sequence no resin flow thickness problem: VF = 0.393
5V	none	-impossible to manipulate the prepreg. -very dry prepreg with black areas	mpossible	separation of the fibres during cutting of the prepreg Material not available for mechanica tests.
6V	low	ply by ply at RT	1h at 140°C 2h at 180°C under 7 bar pressure +vacuum 270 mbar	some first ply delamination during peel ply release thickness OK: FV = 0.624

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APPENDIx F

DETAILED PROGlIESS OF SUSTASK 1.1 MECHANICAL TESTS

(referring to tests matrix page 6 of PE-1102-D/1)

SUBJECT	(residfibt-e code to ‘be tested) x [test temperatures]	partner	progress
~~Comp * 60~~	(~ 1%7, IP,3~,6T’) x [20,120]	~ f f i	1 test at each temperature, extra 1 T rnatc,rial will be provided by AS following problems. f All other specimens in preparation.
comp j (~ ~Qz,02,~60~jS ,	(2T, 4T, ST) x [20>70)]	DLR	~Good transfer of the ies~ procedure: check on 1 T/ All systems with resin 1 are tested. AH others will be avaiIable for PCC meeting in september 94. Need of l)RA resu~fi for 0° behaviows computation.
fwmp 90°		r DB	completed
comp + .45°	(IX, IT,JV) x [20,70,120,140]	IW.,R E	1 W,l P, 1 T,2T,6T, 1 X, IV, I Y, 1 T’ tested. 3T,4T,5T,31~,4V,5 V,6V, 1 T’ results available for the PCC meeting of september 94 (including shear plots, sub routine ready).
tension ± 45°		I D 4S	411 [he tests are performed. Report available for PCC meeting in September 94.
SBSS inledarninar shear	(1 Y,2V,3V,4V,5V,6V, 117,1 T”) x [20]	SNPi3	
DMA neat in s b a weigh[Pr eg g + control	1 W, 1 P, 2T,3T,4T,5T, m - IX,l T,llr,l Y,l’~,]’r” 2V,3V,4V>5V,6V	SNPE	All evaluations completed (availabIe in repor[NT- I 001-S10)
laminates l~loulding ;- C-SCAN	21”,3T,41˙,5”i’,61” 2V,3V,4V,5V,6V	DAS	complelcd, impossibility), [o Ret acceptable 51/ laminated plates fo; testing.
	]T,l V, IT’, IT” IP,l X, IY, IW	DB	Completed
Suh@sk COordi{]a[io~l		DAS	conii[l Llc

APPENDIX F

DETAILED RESULTS OF SUSTASK 1.1 MECHANICAL TESTS

0° compressive strength (DLR)

Plate N r.	icknef	Width{	Strength	Sffain	emp~PMm	w.	:Meari Ssef@	and.Devi	Mean Strai	Stand.Dw.	~Pfate	?Jr. \S@ess 6&	Istrerlgrm o-
	mm	mm	VIPS	0/00									
1T04E	3,17i	6,44[	-47.3,378431-	-24.45S5	~		-_< 6 5_. 6 2 5	12.065581	-252662	1.13-428951	[fio4E	-195.8]	-1934.75023
	3,15	6,45	-507,098791/	.24.0985	RT	/							
	3,18	6,45	-480,863927{	-26.833	R	T _" !							
	3,18	6,3	-481,7411	.%03<	e-l					+ . - — . J			
	3,18	6,49	-480,042;	. Z ; i	l ; i	p - - t			j - - - -	t - + ,			
	3,0	6,5	-52.4]	-26,8	[RT		.512,21	16,66772	-26.467S;	0,470226	II	-795.8;	-21 18.799Fx3
	3.06	6.57	-500 4/	-145.135	RT								
	3	6,55/	'--45s ,9[	-22,765	70]		[-5C.4 ,882	63,614154~	-22.3°5{	0,523Z59	I	.153,03~	.2254,14053
- — - - - T-	J	q	-550/	-22	701								
fT'02E	277}	6.471	-545.6S5}	-27.265	RT	lf7"o2E	-si3.534	22.527907 /	-25..1$875]	<.4004 166\	lro2E	-f%.1851	-2105,27869
	2,73;	6,51 [	-504,155\	-24,49	RT :								
	2,73:	6.52i	-493,674!	-24.71	:RT			i -----					
	2,73;	6,47	_51C.651	.2<.3<	RT	L--J+	- ..i-...	-.-.-;	. . . .	-----/L	. . . . .	. . . .	~-----..-J
1 V 4 E	2,69;	6,461	-394,8!	.13<845)?7	~lT"4f		-502,1333	93,062255	-13,17167	12,84824	1T"4E	-Y85y5'	.2@85 29954
	2,71 }	6,441	-%0,3/	-25,67	RT							[-	'
	2.75/	6..4/	-551,3	0	RT								
	2,69{	6,24	-339,7	-13,35	70		-444,6623	92,822893	-4,45	7,7076261	I	q	0
	2681	6.46	-5(5,'a39	0t	70~								
,1	266/	8.49	-478,W3~	0/	70]								
	2,72	6,47	-424	-23,03~	1201		-424 I	#t)lvfa !	-23.03\	#Wf/O!		01	0
	2,76	6,43	-435,3	-20.40s\	140{		435.3j	mvm!	-20,405j	#CW/0:		0/	0
1P2E	3,05	6,32	-445,6	-10?3+RF	=W2E	.-	+-6t55SSflSi$8452		~~+6fi6@f825514{~		P2E -	+---+56s 65f--f	681Jj374
	3.06\	6,3fi	-442.8	-18>S65	[RT :								
	3,05	6,2	-441	-18,775\	RT j								
	.3m	6,3J	-3'39.5	U\	140{		-303.5 I	#Dhf/01 /	0]	#rev/o!		0{	o
	3,08	6,25	-359,1	Of	120{		-359,1 I	W21vo.	0/	#DIv/o!		q	o
1Y02E	2,88	6,61	-187,9	-3.165~f7T		/IYQ2E	-162,9	23.012823\	-2,8610.	2870975	IIYC12E	-24,61	-683,399
	2,8	6,2	-158,2	-2.62{	RT							/	
	2,88	6,32	-<42.8	-2,595	[RT								
?	2,91	6,71	-84,9		RT						[?		
1V02E	3,3	6,39;	-526.390667j	w2,37(	RT	ifvQ2E	I -5%.47621	f9,1 1104!	-60.57875{	1,6S5674!3	~IV02E	-247.3/	-2090.3577
	3,3	6,8	~ r-4 2 4	-62,15	RT						\I		\
	3,31	6,51	-589%07*	-58,335	RT								
	3,3	6,45	-5W.019	-61,46	RT								
	3,3	6,45	-422.363	-48,73	70		-430,2185	11,109355	-47,88	1,X@mFsji		i—t97,14j	-15K611
	3,32	6,47	-438,074	-47,03	70								
	3,32	6,46	-426,628	-46,075	120		-417,2145	13,314114	-44,7875	1,82081{		-143,8S5\	-1764,%<1
			-e0,',0	-43.5(	f2131						II	!	
1			-1129,1/	.31 ,83]	RT	+1W02E ;	.990\	120.70051	-25,23	9.33360	W]\two2E	.i94.i71	-4969.%44
	3,32	6,2	-9\2,91	-16.63]	RT							[	
	3,33	6,3	- 9_2 8 {		RT								
	3,37	6,36	-584.1	0	120		-584,1	#DIV/0!	0	#DIV/0!		0	0
IX92E	2,Q(	6,S1	-282,971			[IX02E	I -265.33/	24.946727 \	-6.585]	KNV/CII	HX02E	-72,74(	-f22E.27$2
	2,9i	.S,39/	-247.691	-6,5851									
	2,8	6,32	-248.25 \	-5,73!	7GI		.2$$,7691	0.7339768[	.5,?7251	0.0601041	/	.60,031	-~1 S2,463
	Z,V1)	o,-]~	-L49,288!	-5,8151	7 a J~						i		
;T21D ~	2,86;	6,51							"" T"?,-q"c<	85539	12"T"z1 0	:!?~y?s]	-2032:80>
	2,64	6,49	-454,654	-15,31									
3T22D1	2,91	6,41	-486,782	-23,715		3T22D1	-463,577	4,1082904	-23,6875	0,0388809	3T22D1	-142,38	-2191,36
	2,96	6,42											
	2,96	6,42	-480,972	-23,65									
4T23D	3,86	6,52	-164,102	-7,345		4T23D	-135,404	42,443311	-7,395	0,2832149	4T23D	-11,12	-908,414

""-HBE=l==!12d'.gg"=~-s S---" ---"

37>4 ... _JJ3 _- >

= I = L = = I

k + a *2 o+$iii=-=+==q---

/ 3,55 6,461

- - - - - 1 - - -~ + f ~ ~ - - p_ = = - — - + ~ - - : - - — $ - , - ~ - - - - a

~~~ ~=,ii=:d..i=:i:iiji -.-.,.=-. Z Z

* F 'z: 1 , ~ — — = i q " " '-'-'; f i Y i 7 2 % * -

2,85 6,42

- = . . $ = . . _ . . _ _ . !_~—— . —' .---

""-:?6""e7-5~ ! —"; "" 'T '" :

I "-'"~ ?;:I"";.yi:,'" ""='""+ t,
~~~

“ m

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3REz+n92-0314

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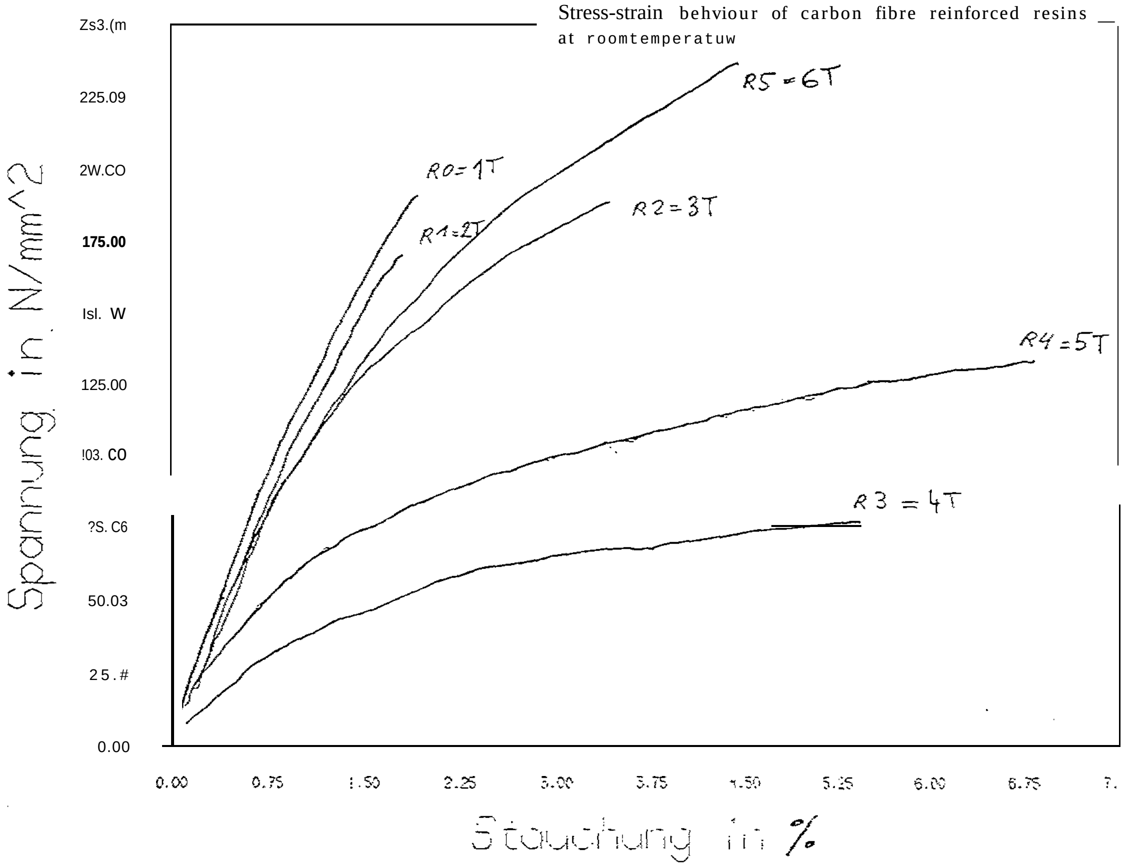
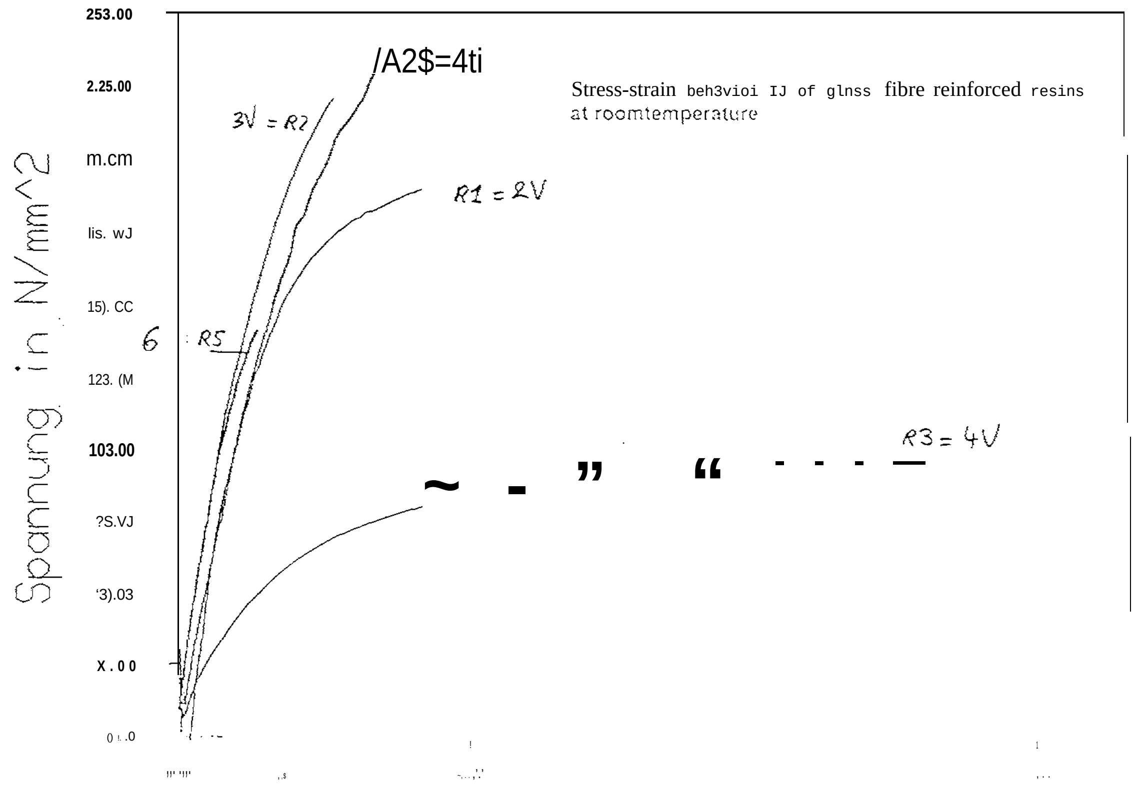
APPENDIX 1?

90° **compressive behaviour** (DB)



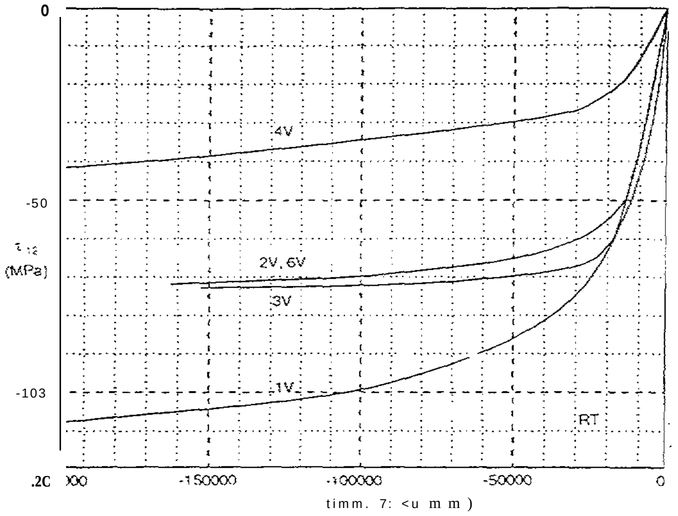
Stress-strain behaviour of the carbon fibre reinforced resins [illegible] loading at roomtemperature.



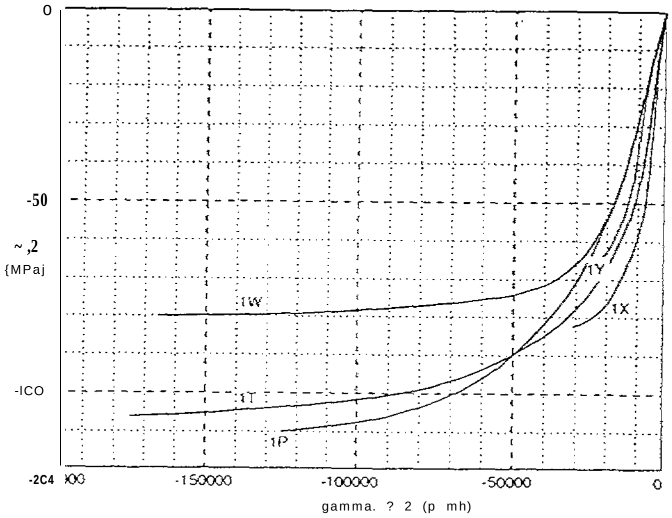
Stress-strain behaviour of the glass fiber reinforced resins subjectso@ transverse compression loading at roomtemperature.

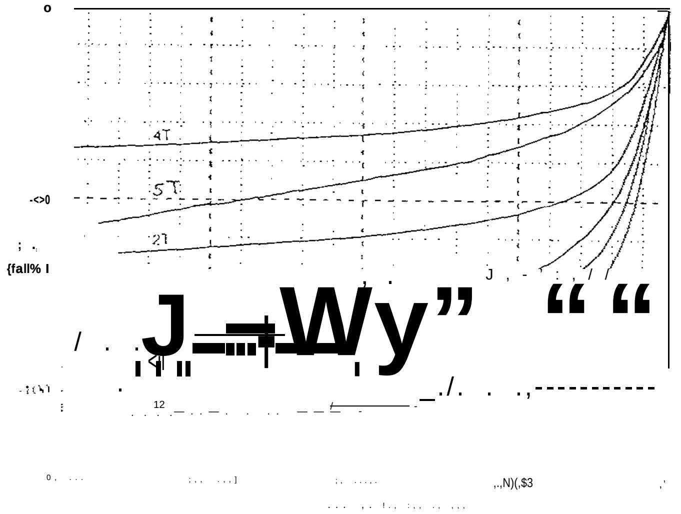
APPENDIX F

(f 45°) **compressive behaviour** (NLR)



Effui **of few** type m sk?ar curve fof E@ass **cowesite**

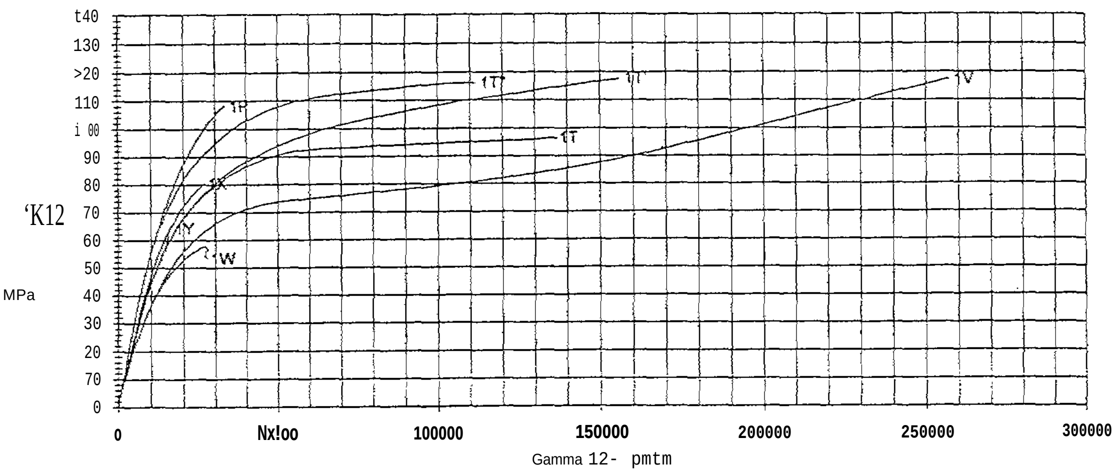




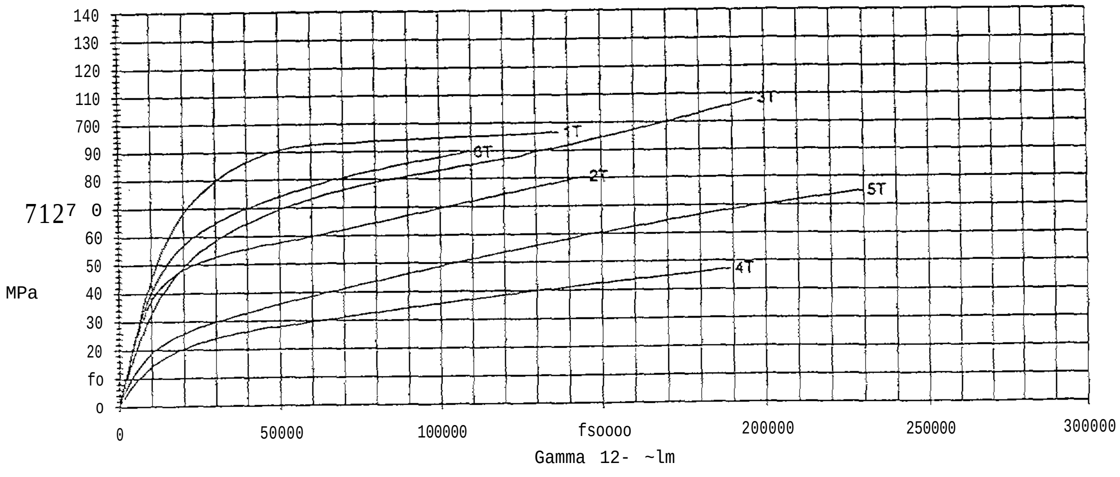
APPENDIX F

(f 450) ten~iie behaviour (Dassault)

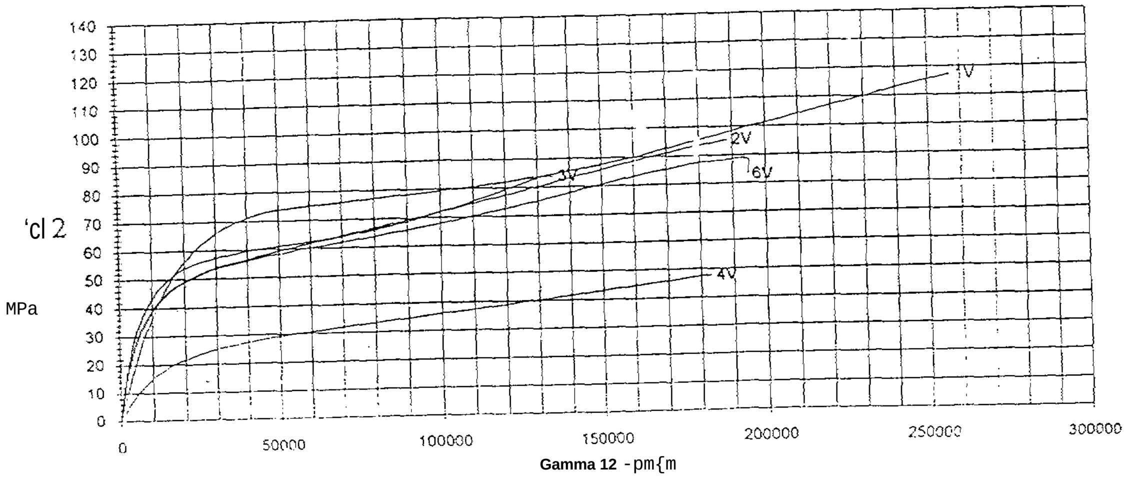
Reinforcements effects with resin 1 (DAS081 at room temperature



ReSin effects with carbon fibre (T300J at room temperature



Resin effects with glass fibre {Glass E) at rOOm **temperature**



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APPENDIX G

SNPE MATERIALS ELABORATION

FRONl DELIVERABLE REPORT (except appendix and figures)

(NT-1001-S/0} distributed to all partners

SNPE

“rN„4 7/941 cRB/fM-FPJDR
51 FFUS[ON RES’I%HNTE

I - H’$TRUDVCTION

In this pal *of the research* @ask i) the cmntributiop of SNPE consisted of:

- elaborating resins, trying to rnexx the required rnechani=l *propmies defined al* the kick off Meering
- getting a supply of nwst of raw rnz(eriais,
- producing and providing prepregs to the partners concerned by the manufacturing of Iamimtes,
- providhg neat resins ; uncured resins and piates.
- mak%g various evaluations and measurements on the prepregs, on the resins and on U.D. laminates.

As the evolvements of t3ese works were more or less overlapping, for the sake of ckr-ity we will repxl successively on the **resins** : formulation and pro~rdes, then on the short beam shear strength (SS3S) results on U-D. laminales and last on the materials supplied to the partners.

2- RESINS

2.1. AX~ Of ~Kh - Background

Tire axis were defined with *partners* and particularly with DASSAULT. They are recalled in annexe 1 (copy of P. 5 and 6 of MN-lCK)l-D/1).

As a reference case Ro, resin DA 5C18 was chosen. It is an industrial resin which has, among other propem-es, controlled flow for making the iamktate and relatively high Tg.

From a practica} point of view, ii was **availabie** and could be used making the set of prepregs with the various fibres of sub-task 1.1. during the [i me spent to think and to ela~mte the experimenml resins.

For resins RI - R2 and R 5 let us fi ml recall some properties of thermoses

The propenies at **break and the** thermal propezlies of vitreous networks are difficult to co rreiate w i[h I he s(mcmre of the network because numerous factors act simukanmmsly : cross-linking density, aromaticity of the framework, flexibi]i[y of the pivot, mol&u\ar cohesiveness.

The data we have gathered and our own experience suggest that the cohesion is a factor rather more important than the cross-linking density : the more the system is cohesive, the less it can absorb energy by viscoelastic and plastic processes before break.

An increase of the cross-linking density gives a tendency to go from a ductil

EE!EE!3

For tie thermoses epoxy-a-nines, the elastic propalies (Young modulus) are generally little de+wmdeni of the cross-linking density and are mainiy drivem by the cohesive energy and qeeiaf~y by the par&of it associated with intermoledar hydrogerr bonding.

For filled resin (R4 compared to R3 j the aim of a positive influence of fillers to increase the matrix yieki strength would need, in our opinion, much remrch.

A basic requirement for the filled ma[rix in the composite is that the panicles have to be much smaller than the distarwes between t%res in the mmposite, which we of the order of the diameier of the fibres, currently 5 to 8 t-.rm for the carbon fibres.

For compmds with short fibres it is weii known lhal the reinforcement effect is be[ler if fibres are above a minimum size.

it is known , from the literature ‘, that adding of mineral particles kad 10 an increase in modulus and this, tie belter the smaller the particles.

In the past some tiab have been made in our laboratory.

- with a resin whose ini(ia[t@rsiIe properties are :

  E = 37CDMPa - u = 71,5 MPa, ande = 2,5 %.
  The adding of a carbon black about 20 nm gave ::

  for L5 ~r E = 5545 MPa, o = 33,9 MPa e ‘== 0,8 %
  for 30 pee E=5800MPa, cr =25 MPa& == 0,5%

There is an increase of the modulus but a strong dedrease of (he ultima(e stress. There is ***besides, a*** huge thixotropic effect and the restrl[ing very high viscosity makes very difficult the mamsfactory of safe tensile samples.

---

* FOR FX.mrw t% - RIJ XU, TAKASHI NISHlh’0, M.!() KATWHIKO NAKAMAE W-)LVMF.R (1992) 33, i“ 5167.

- with a very ductile resirr whose initia[wakes are :

  E = 15C0 MPa

  - yield stress G ⁼46MPaforc= 9%.
  . s[re$s a br~ G = 46 Mpa for c z 15 yo

  adding sitiea pariicles, abou[50 pm diamtier, gave :

  E=3C00. MPa, o=48MPa for&= 5%

  a big increase of (he modulus, and the small increa% of the sm.ss are to be noted.

- Mo~e rexmly some trials have been made with whiskers of diamem 0,2 10 0,5 µm, a mean length of 10 to 20 µm and Young modulus of 280 GPa. Starting with a resin with E = 3900 MPa, σ = 82 MPa for ε = 2,4 %, adding of these fillers gave:

  . with 15 pcr E = 4700 MPa, σ = 47 MPa for ε = 1,1 %
  . with 30 pcr E = 6000 MPa, σ = 47 MPa for ε = 0,8 %

with a good enough processability for the 15 pcr and somewhat at the limit with the 30 pcr.

2.2. Manufacturing of the experimental resins and results

To meet the required mechanical properties, 9 experimental resins have been formulated and then their tensile properties measured according NF T51-101, from which 5 resins have been selected having nearly the required properties.

At this level of the research the other main constraint is, of course, feasibility tests of both of resin plates and prepregs, for [illegible]

Before mechanied evahation a]l resin samples were left 16 H at 150° c to try m release the Wrssions coming from the machining.

The mechanid properties at 23” C of f-he reference resin Ro and of the s-sleeted experimental resins RI to R-5 are presented In table 1 and illustrated by typiezd tensile

In the tabie are mdmateA E, !.he maximum value of u {in nrosl of ease at break), s

Referertee resin Ro (figure 1) is a relatively brinle resin and there is much *scatter on the* vaiues of ttre rsi[imate stress o, elastic modulus E is r-mher high.

Resins R 1 and R2 (figure 2) meet the requirernen[El ~ E2 and G 2> c i..

R2 is a simple epoxy-amine sys~em, R 1 is a non stoechiome[ric epoxy-amine system with a small amoum of rubber (CTBN).

To my to ger an ‘mcr- with “the imroduction of fillers ‘m initial resin R3 was eiabora[ed with a very dueiile behaviou r : R3 has a low modulus, iow maximum stress rvhieh is reached at about 6-7 % strain, the stress remains nearly constant till the ultimate stm.in at 23,9 % (wi[h a noteworthy small scatter on the va}ues].

AS fiilers to add *in* R3 to make R4 we have imrodueed [he whiskers cited in 2-1.

For R4 (fig. 3) the modulus is somerwhal higher than for 53 (~ = 1,25)

and the ultimate s&ess much higher (~ =1.4). We think that these results have to be attributd mainly to the very high ductility of R3 associated with [he form fx[or of [he whiskers.

R3 has a complex formula, with di functional and tri *func~ionnal epoxies, amine* hardeneI and a higtr proportion of CTEH’J which constitute probabiy the eominuotrs phase of the *resin.*

R4 is basicalIy R3, bu[with an amount of 5,9 % VOI of whiskers added. Note that in an otier trial with less whiskers added (3,5 % vol) we had, with respeet lo R3, no increase for E but yeI a signifieative irrcr=se for G (5 I MPa) assmiated wi!h a redueed strain.

Resin R5 (fig. 4) has an ukimate stress higher dran ail other resins RO to R4 Thk resin is a simple epxy-amine system;

remarks

1. On fi[lers

   Making the prepreg and then the iaminafe, the risk with fillers is to gti some segregation, and more es~iaHy wi[h whiskers (or other aniscxropic p~cks) 10 get some aniso(ropic effects.

2. On tensile tcSs

   We think that the value of σ can be handled with confidence (≡ intrinsic material property) when the standard deviation is small compared to the mean value. When the standard deviation is high, that means that the result is also somewhat dependent of the "defects" of the sample being evaluated, which have an effect when the K_{Ic} of the resin is weak (≡ brittleness of the resin) and that the measured σ is lower than the "true" σ of the resin.

2.3. Thermo mechanical properties

2.3.1. Dynamical mechanical thermal analysis (DMTA)

The resin samples were tested in flexion on a Polymer Laboratories apparatus at a frequency of 0.3 HZ and with a temperature variation of 3° C min^{-1}

The apparatus gives the curves log E' and tg δ = $\frac{E''}{E'}$ versus

T1.e results are summarized in table no 2 where are presented the ~emperature of vitreous mnsi[ion Tg defined a 1 at tie maximum of the peak of the tg 6 curve which is the more preci= and reproducible determination to wmpare r~ins and b [oc the Icg E curve, al the onsef point which is a more realistic value for I.& of the resins.
With these d.~ the maximum testing Iemperar, “re for experimental resins code 2-4-5 (RI, K3, R4 here) was limited to 70° C @lN-H20-4-D/0).

2.3.2. Variation of tensile properties with temperature

The resdts for [he temperatures indicamd in !he program of task 1-3 are presented in able 3 and figures 12 to 14 ; they are also to be compared wilh those al ambtat tempemture.

The general trend is a *dezrease* in E and G with increasing temperature. This dwr= is im@@rt if the temperature of the rrreasrrrenrat is near enough the Tg tempecmure (onset mint) and is in qualitative agretmat with the log E carve in DMTA : it is the case for Ro at 140” C, R5 at 120” C and R3 and R4 at 70” C.
The exceptions are Ro at 120° C where c is higher than at 70° C and Rf at 70*C where u is higher than at 23° C. For the 2 cases there is less sca[ier on a and am incrase in -e as if there was somtiing like a transition briuleductile.
For R3 (figure 13) ihe rnax value of o is reached at & between 4 and 5 % the value of s = 19,4 % given is this permitted by the extensorrreter *range used erd is nol the* ultimate value. For R4 (figure 13) Ihe max wdtie of a is reached al E between 3 and 4 7. and this “’resin” becomes much more duclile than at 23 °C.

For R-5 at 120° C, 2 xts of vafues are given in tab[e 3. For this resin, *at* 120 ‘C, there is inhomogerreous strain and nectirig phenomena which iimits the max cs
The first set of values is for all the samples (*n* = 6), the somnd for a seleelion of the 3 s?mples with fhe higher E.
For afl the specimen the cuwes have [he aspect shown figure 14, for which the final part (af~er c max) has not to be considered tree.auw it *comes* from the shnnbge of the pan of sample under the extensometm while the necked part becomes thiner and longer under the moving of the cross piece of {he tensile machine.

msNPE

N.T. N“ 47/94/CRBIM.FP/DR
OIFFUS1ON RESTREINTE

3 - *SHORT BEAM SHEAR STRENGTH* *(Sutmsk l-l)*

Measurements wexe made according RO. **4585 (TW-1 04)** for glass fibres and T 57-303 for the others.

I.-@ us @l that SSBS is dependam of matrix sh=r propeuies, matrix fibre irrrerfaee and laminate perfection;

3.1. **Results** on materials based on reference =in DA5d8

The eornpkte set of data for samples of the programnre was squired on UD laminates made at SM?E except material. 1 T' (with high fibre eoruent] made at EM&4 (I@ and is presented in Table 4a. Data for IT and other samples from DB are presented in Table 4b.

hi rdie 42 results are given with aiso sample thickness e and ea!cuiamd % fibres.

AI ambient temper-mm very good values of SS3S are obtained for lT, 1 V, IW. For DB material values are samewhat {ower bul still gti.

Mluenee of the type of carbon fibre (IT, IX, lY) : we find in Qbles 4a and 4b the wel[known belraviour csf decreasing SSf3S with fibres of incr~sing mochdrrs (corresponding to higher tempera[rms manufacturing).

Mkrr~ of surface irea(rnerr[of cation tibre T 300 (IT, 1P) : ma+eriai with untreated fibre gives, as waited, a Iower value.

lnfluerree of VO[percentage of fibres :

There is rm definitive conclusion because varia(ion is aimos[witflifi the ~t[er. The tendencies one would look for are con[rariiclory : for SNPE lamina[es (tT, 1 T“) there is an increase of SSBS with fibre corr~ent but it is a dezrease wl]ich is observed for DB laminates *(IT', JT, 1 T“).*

Influence of temperature :

Whatever (he material, SSBS is decreasing wi(h [en]& mture ; the classification of materials according SSBS al 23° C remains tz same at higher ~empaatures.

3.2. f7em11ts on ma?erials based on ex~rimenfals resins

Results on experimenrd laminates made al SNPE to check the feasabili~y are presented in able 5 a and first results on Iaminates made by DASSALJLT in rable N)
None value reach [his goI wi{h reference resin.

4- *MATERIALS SUPPLY TO PA RNERS*

Much work has been **done to** comply the materials requiremmts expressed *by* the Ieaders of the wor~ng groups, with for subtask 1-1. prepregs by smal~ quantities (D=uII - PE 1102-LM2), for swbtask 1-3, fibres, uncured resin, neat resin plates (ON ERA PE 1301-0/3), for subtask 1-4, prepregs @HJ? - PE 1401 -?W3).

The chronology of the deliveries is presented in Annex 2 where aiso are recaik=d the cure c@eS which can k appIit?d to resins and prepregs.

. F'rcpregs were manufactured on industrial. pilol or laboratory facilities. deperrdem on the materials and the quantities. With the sending was joined the rrrxessary informations to make the laminates : surface mass of the prepreg, surface mass of fibres, pementa.ge of **resin,** jf the case percentage of volatile, @nmoI ~d k=Ji=iion of def=~ and a cure cycle, either weli established as for reference resin, or indicative (not optimized) for expaimenkd resirrs.

- with uncurti resin was dispatched an information note with a cure cycle.

. Piates thick enough co machine 3 mm thick sarnpies were providd. We have *been* abie to make safe p!ates of resins, which is no: an easy thing, particularly with DA 508 which is optinriti to make laminatti but not plates or blocks. For experimental resin plates, a sample of each has been tested by DSC to verify that (he thermoset was complelel y cured.

5 - CONC13SIONS

The work devoted (o SNPE within rask 1 was :

. for[muiating resins wi[h specified mechanical propenies.

· making various measurernen[s : tensile propcmies and DMTA on resins, SSBS ofi U.!2. laminates.

· Providing partners wi(h materials : fibres, uncurd resins, nea! resin plales and a great number of various prepre,gs.

Next work will begin in optimizing resins according the resul[s of compression tesls of partners.

DOC. DGT/'DEC/MT N" 59.211 35
PR-ooo3-D/l
October 4th, 1994

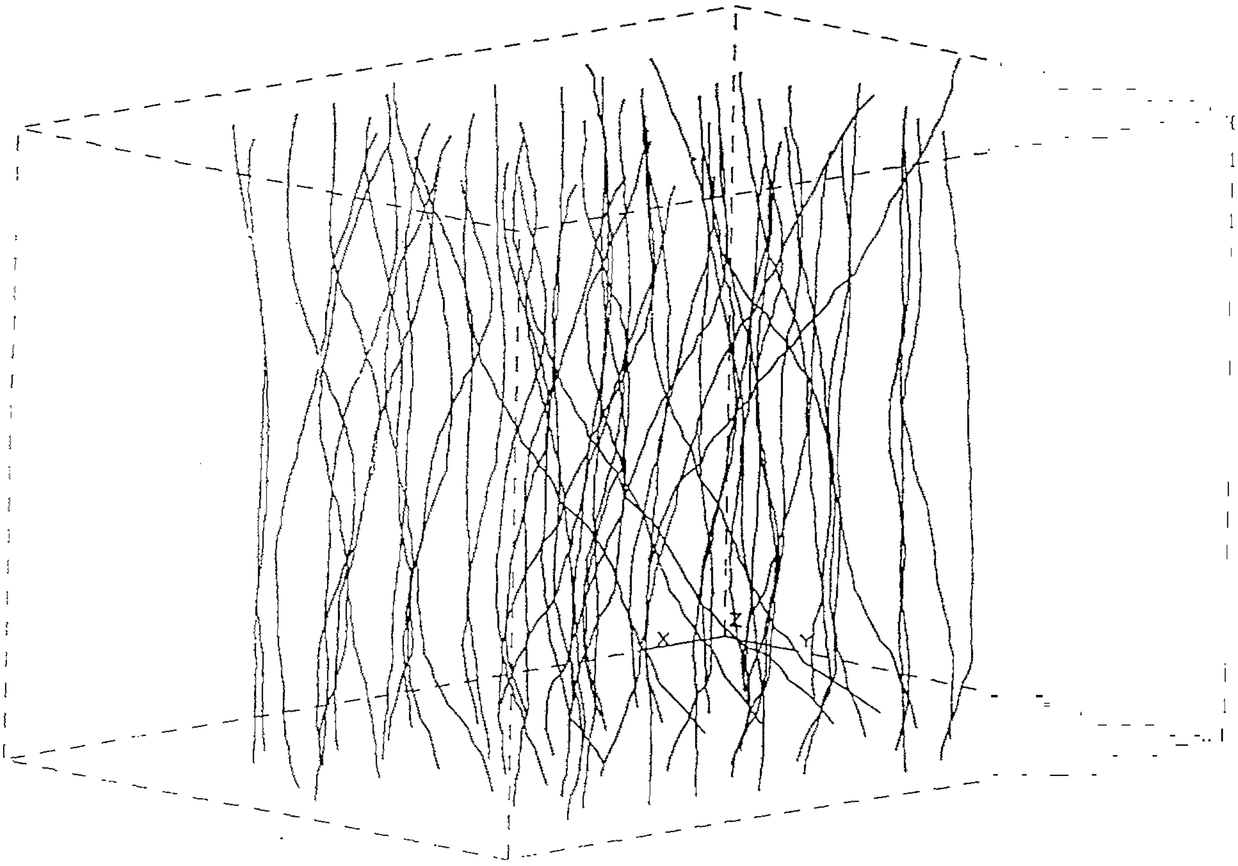
APPENDIX H

FIBJNLS INITIAL MISALIGNMENT ASSESSMENT
"Presentation of the method"

One of the main parameters determining the compression stren@ of composi laminates is fibre n-iisaiignement. At present ordy limited work has been concerned with measuring th parameter, for example the influence of the number of consecutive plies on fibre rnisalignement is n known.

During subtask 1.1 the fibre misalignment will be assessed by the followin experimental technique (ONERA). Initally the sample will be polished into 30 to 50 cross-sectio approximately 20 ~m apart. The precise number of cross-sections depends on the nature of the fibre. Ea cross-section is examined by optical microscopy couples with data processing in order to extract t position of the fibre centre. The fibre paths are reconstructed through all the cross-sections from which t sample is analysed for fibre **misalignment** and waviness.

First example of T300 fibres arrangement in a unidirectional k.rninate :



APPENDIX I

Tables and figures of $4.3

PROGRESS IN SUBTASK 1.3 ABOUT CONSTITUENTS CHARACTERISATION

Compression of embedded filaments with LY556(27% HT9~2)

Strain range of first failure

Fibre	strain range without correction	strain range with correction
M40	0,1?%-(),75%	1 ,~7yo-~ ,65%
Bore	1 ,3~°/ o'l ,~1%	2,200/o-2,8 10/.
T300	3,05yo-3,~~°/ o	3,95 °/0-4,61%
Verre	8,24?0-26,1770	9,~4?&-27,~7°/o

Table f.

Strain range of saturation

Fibre	strain range without correction	strain range with correction
M60	~t71?!o-1 ,700/Q	1,61 %-2,60Y'
M40	0,9~~o-3,58%	~ ,80yo-4,48°/o
T300	4,8~°/o-5,4 ~"/o	5,750/o-6,330/o
Verre	8,750%-42,50"/.	9,65%-43,4 (1"/o

Table 2.

Brite Euram bop Tests (mean values)

fibre	**Diameter pm**	**Failure Strain %**
T3U0	7.31 S.605 @8.30%)	2.01 tO.18 (*8.95%)
~ntreateci **T300**	**7.48 tO.33** **(f4.44"4)**	**1.97 MO.40** **(t20.61%)**
M40	**7.34 tO.81** (*11.1?%)	**1.02+0.13** **@12.43%)**
M60	8.24 iO.56 (31 0. 74%)	1.ootO.13 @3.1%)
E-Glass	22.42511.71 @7.65%)	2.67 i0.21 (27.8 1%)
Boron	136. Otl.99 **(i I.46%)**	0.59 **i-o.04** **(+7.0%)**

Table **3**

Resin	E tGPa}	c {Ml?a)	**E (%)**
RO - DA508	3.6 @.3)	63 (7)	1.8 (0-2)
R I	2.6 @.2)	61 (7)	2.7 [0.4)
KU?	2.56 (0.07)	90 {4)	7.2 (0.8)
I?3	1.76 (0.06)	40.3 (1)	23.9 (0.7)
R4	2.2 (0.16}	56.0 {1)	6.2 (0.4)
R5	2.81 (0.03)	99.3 (2}	8.1 (1)

( ) : standarcl cieviation

Table 4 Tensile properties of reference resin RO and experimental resins RI to R5

	Room Temperature	Xlac	120”C	140”C
RO	1,470 (0,046)	1,407 {0,133)	1/334 (0,096)	1,172 (0,015)
R I	\ 1,228 (0,02’2)	1,056 (0,012)		
R2	I 1,274 (0,023)		0,994 (0,013)	
R3	(?,/303 @,070)	10,422 (0,014)		
R4	1,066 (0,125)	[0,534 (0,056)		
R5	1,216 (0,089)	[	0,959 (0,018)	

Table 5 Iosipescu Shear Tests
initial h40dili ((lPa) versus Temperamre
Mean **VaiLie** and (Swdard Deviation}

Fibre	L_c (µm)	$\sigma_r(L_c)$ (MPa)	τ_m (MPa)
T300-50B	365 (18)	6495 (40)	60 (3)
T300	455 (14)	6316 (24)	47 (2)
M40-50B	297 (6)	4943 (7)	52 (1)

Table 6 : Fragmentation results in the coaxial geometry with the DA508 reslrt .
Mean v~lue and {standard deviation].

. . .	T = llO°C and v = O,ozcrn.s-l	T = 80”C and V = t3,04cm.s^{-1}	T = 80”C and V = 0,t)8cm.s^{-1}
T300-5oB/ R l f LY556 27XXOHT972	L_c= .532 {58} pm or = 6192 (88) Ml?a $\sim_m$= 40 (5) MPa	L_c= 474 (42) w or= 6280 (68) MPa ‘Im = 45 (4) IVLPa	L_c= 405 (4) p-m or= 6404 (9) MPa TM =53 (0,6) MPa
T300-5oB/ R2/ LY55627’XOHT972	L~ = 635 (100) pn-1 or = 6059 (118) Ml?a TM = 33 {6) MPa	L_c= 476 (53) w $\sim_r$= 628CI (95) MPa Z_m= 45 (7) MPa	L_c= 624 (100) ~m ~r=6070(122) MPa Z_m= 33 (6) MPa
T300-5oB/ R3/ LY556 2T!JoHT972	L_c= 481 (47) ~m or= 6269 (77) Ml?a -c_m =44 (5) MPa	L_c= 493 (83) p_rn ~r=6254(133) MPa TM = 43 (8) MPa	L_c= 469 (19) ~m ~r=6287 (33) MPa TM = 45 (2) MPa
T300-5oB/ R4/ LY556 27’ZOHT972	L_c= 421 (74) wı ~r=6382 (136) MPa $\sim_m$= 52 (10) MPa	L_c= 450 (30) pm 6r=6320 (54) MPa Z_m= 47 (4) MPa	L_c= 491 (45) pm or=6252 (71) MPa $\sim_m$= 43 (4) MPa
T300-5oB/’ R5/ LY556 27 °/oHT972	L_c= 667 (210) pm or=6036 (244} MPa ~m = 32 (11) Ml?a	L_c= 558 (124) pm ~r=6165(167) MPa $\sim_{111}$= 39 (9) MPa	L_c= 497 (66) prn ~r=6245(100) MPa TM = 43 (6) MPa
‘T300-50BI DA508/ LY55627°/oHT972	L_c = 365 (18) µm σ_r=6495 (40) MPa τ_m = 60 (3) MPa	▨	▨

Tabie 7: MuItifragIlleIltaliOn res~lits ill the coaxiai ‘geO~netry wiih the T300-5oB/R*/LY556(27 °/0[3T972). -
Mea II value and (standarcl dcvifiti{}n).

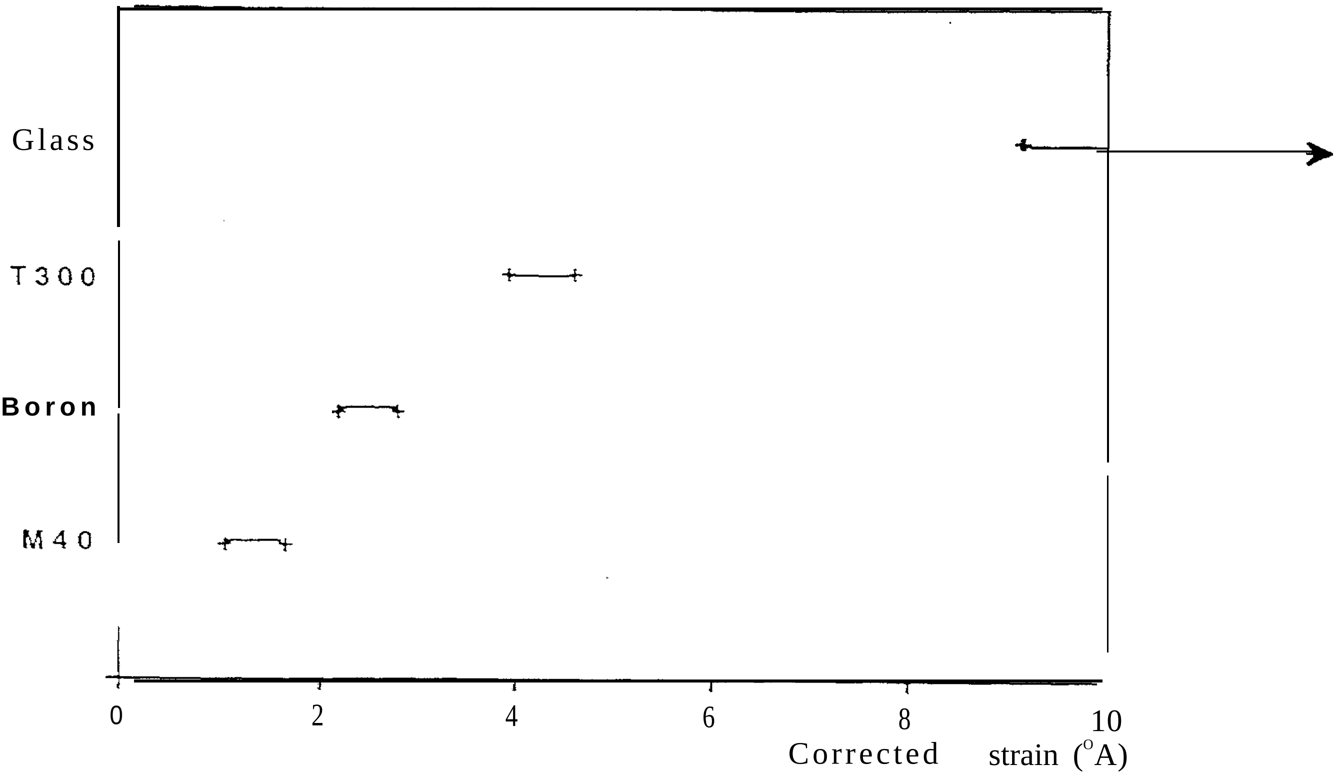
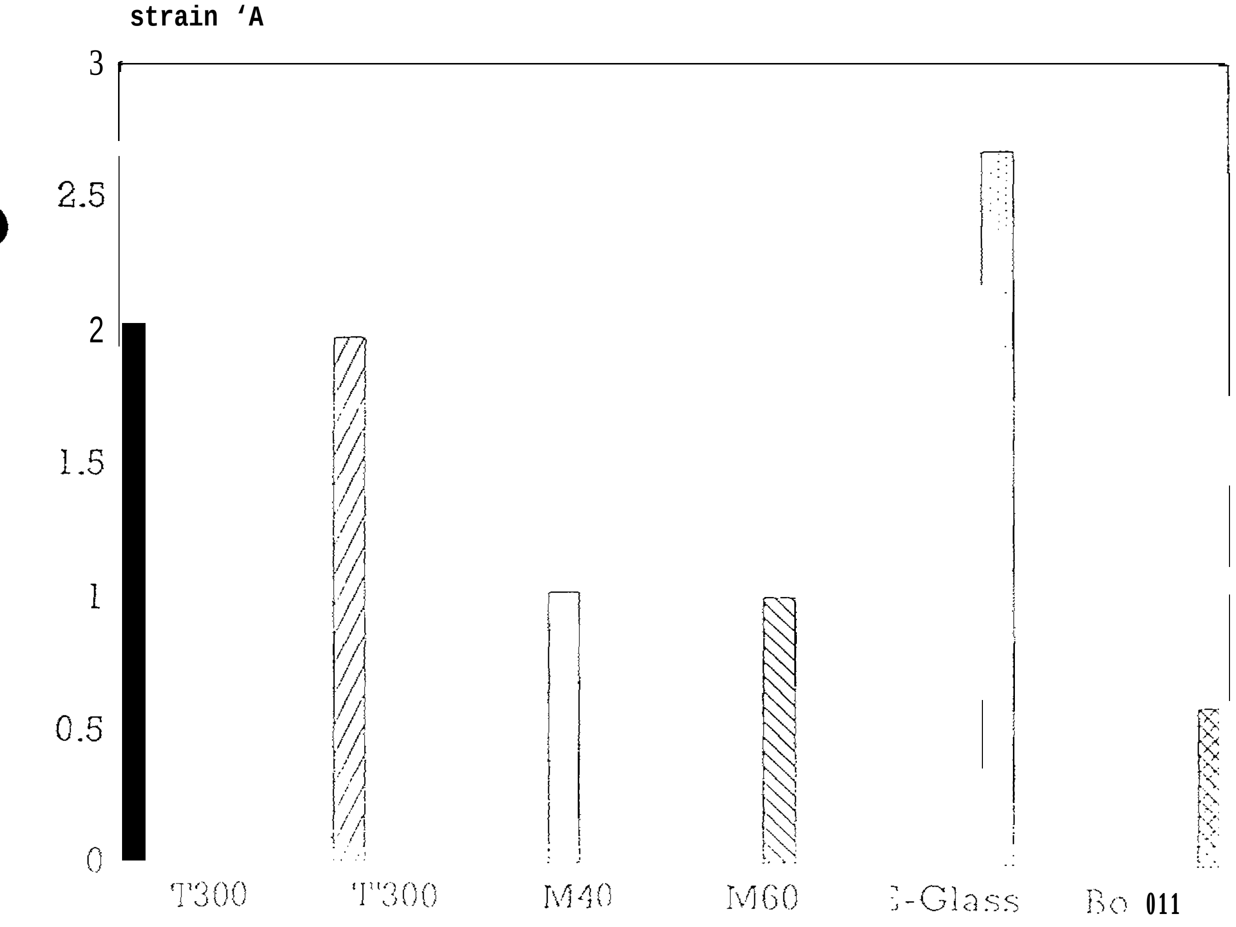


Fig. 1 : Compression of embedded filaments ; strain range at first failure.



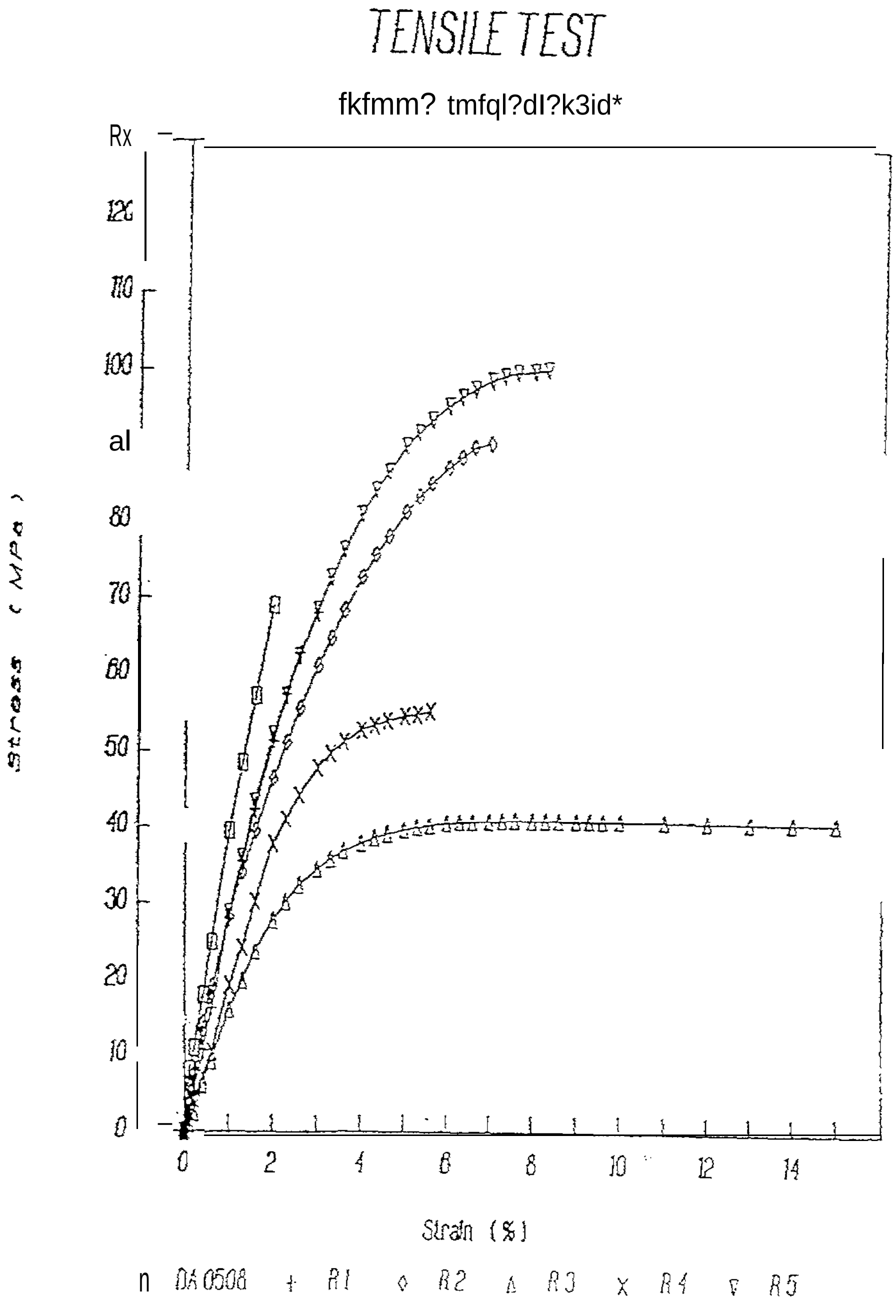

TENSILE TEST
Stress (MPa)
0
10
20
30
40
50
60
70
80
100
110
120
0
2
4
6
8
10
12
14
Strain (%)
□ DA0508
+ R1
◇ R2
△ R3
× R4
▽ R5

Fig. 3

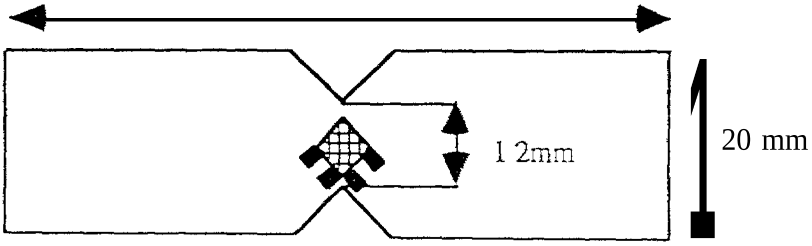
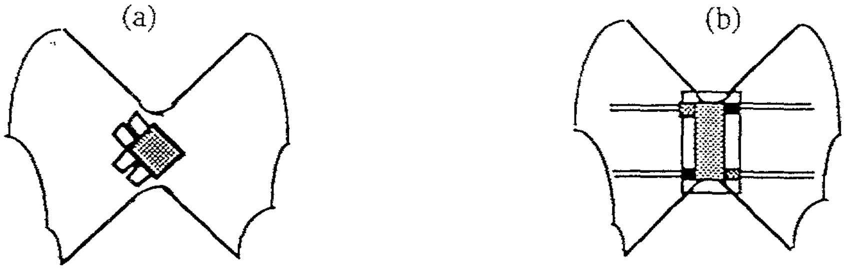


Fig.4 : Iosipescu shear specimm,



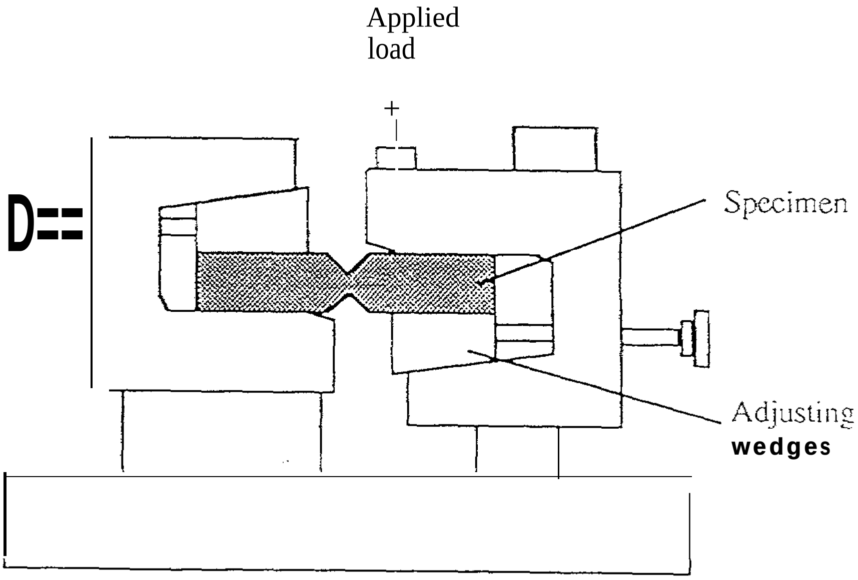


Fig. 5: Strain gages used iil study (a) conventional 0/90 stacked gage,(b) full section in slacked configuration.

V-[lotciiedkanl test specinwit [lwuntwl in test tixlurc.

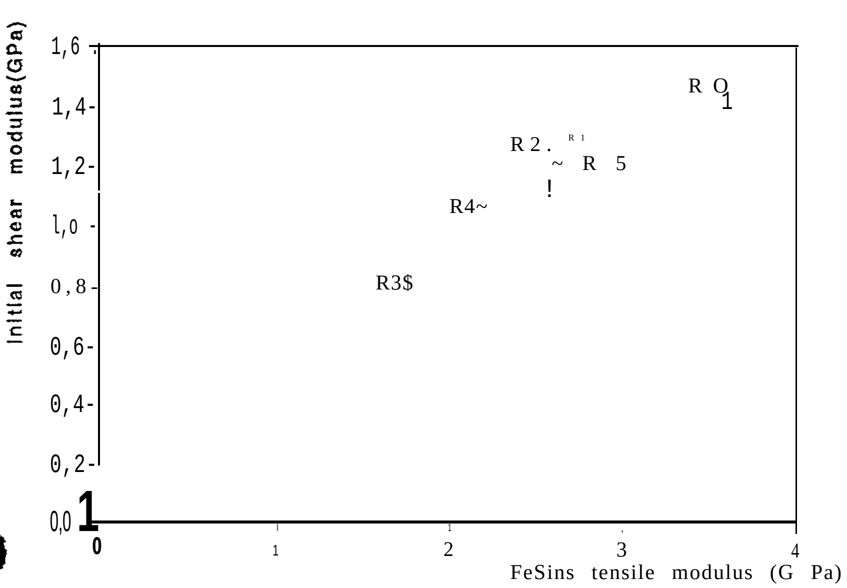
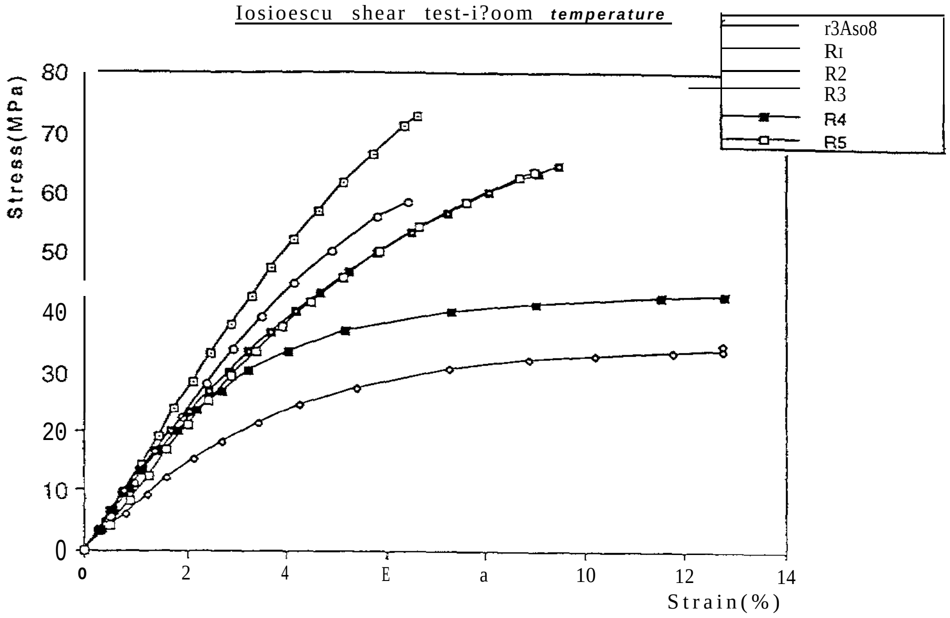
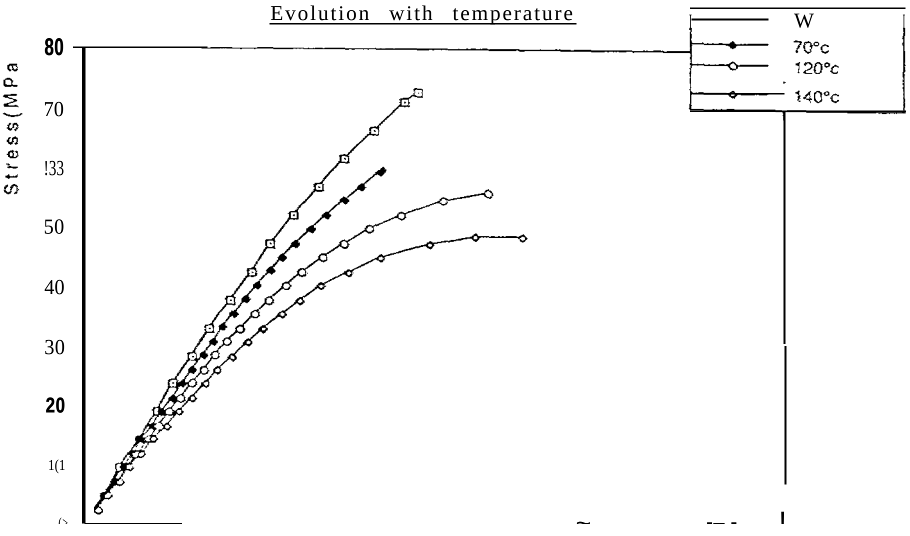

losl~eseu shear test
Initial shear modulus(GPa)
1,6
1,4-
1,2-
l,0 -
0,8-
0,6-
0,4-
0,2-
0,0
R O
1
R2.
R1
~ R 5
R4~
R3$
0
1
2
3
4
FeSins tensile modulus (G Pa)

Fig. 6


Iosioescu shear test-i?oom temperature
Stress(MPa)
80
70
60
50
40
30
20
10
0
0
2
4
E
a
10
12
14
Strain(%)
r3Aso8
RI
R2
R3
R4
R5


iosigescu sheaf test with C) A508,
Evolution with temperature
Stress(MPa
80
70
!33
50
40
30
20
1(1
W
70°c
120°c
140°c

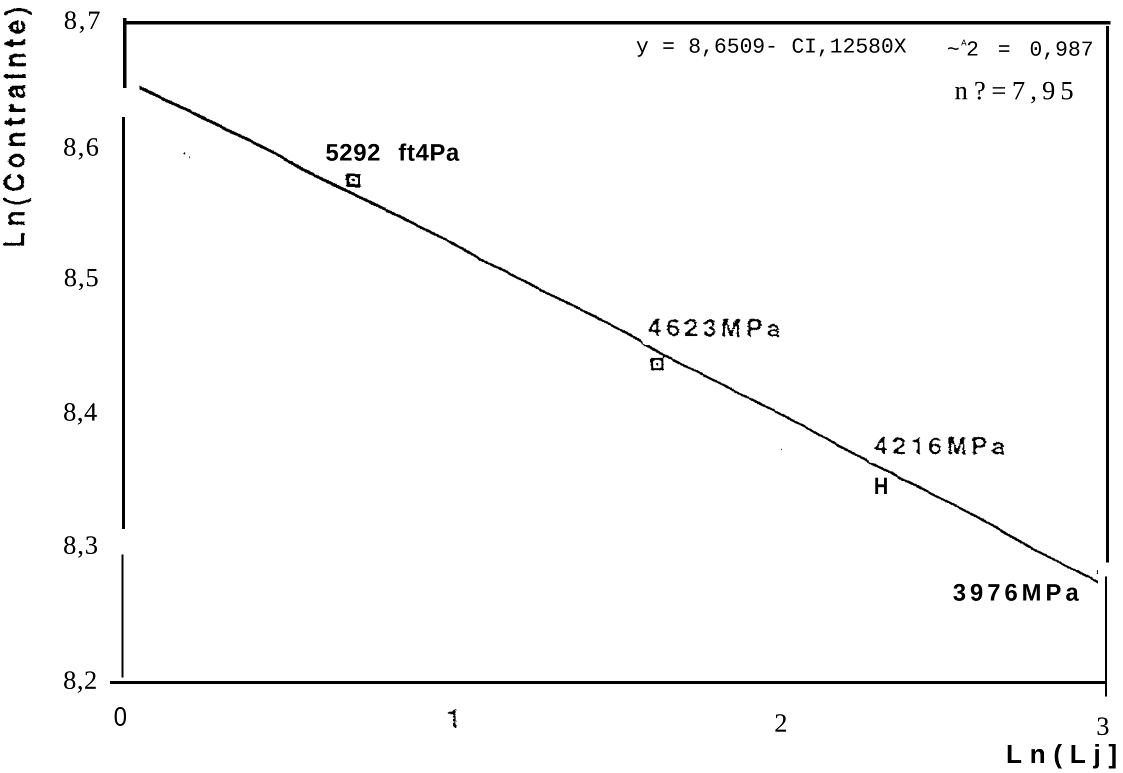
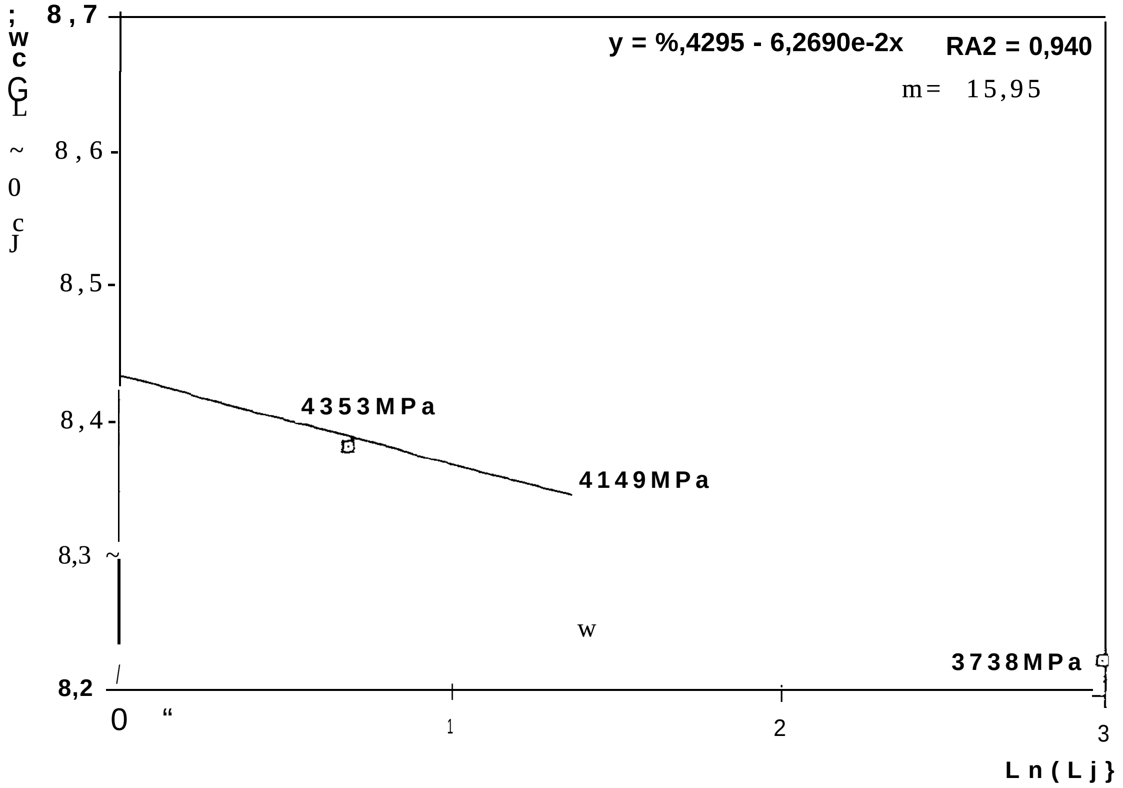


Fig. 7 : Tensile tets of single filaments T300-12K-50B.



Fiq. 8 : Tcrls; le l(?St S 01 single filomenls M4('I-12K-5C)E.

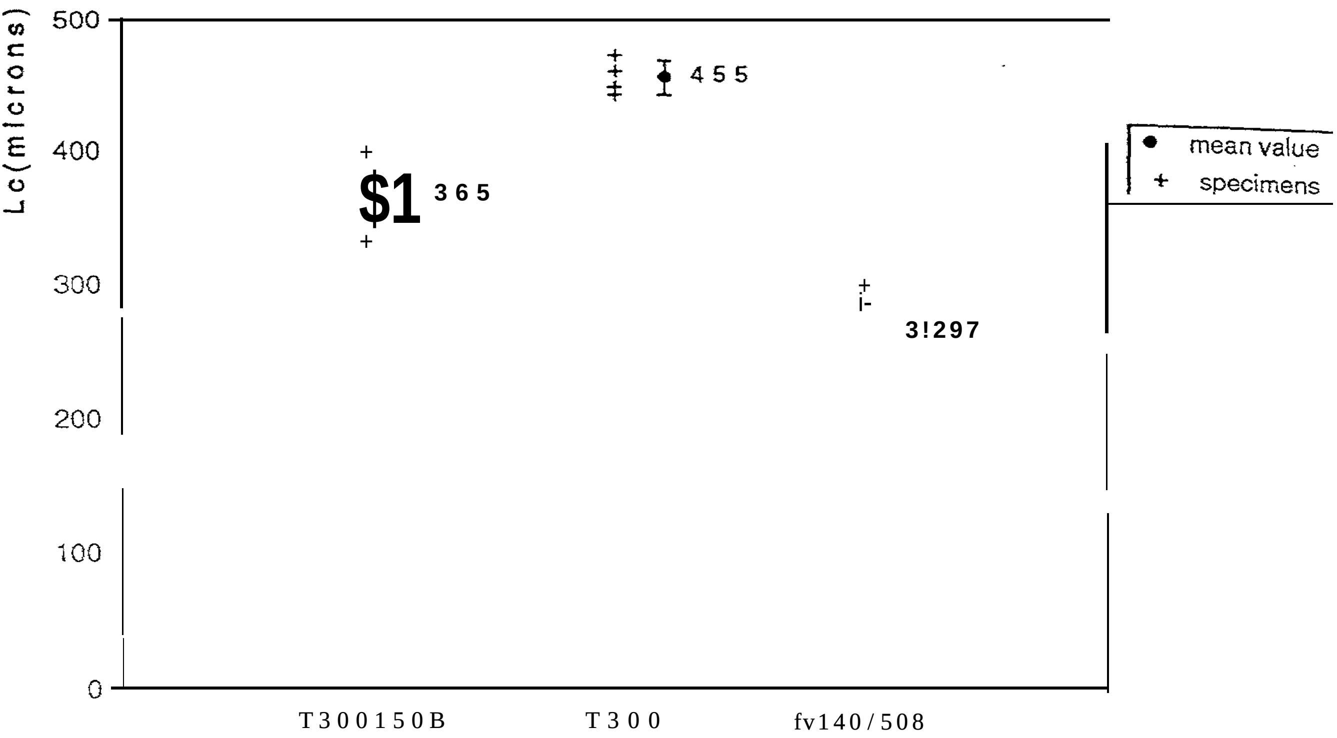


Fig.9 : Tensile tests of embedded filaments with the 13A508 resin.

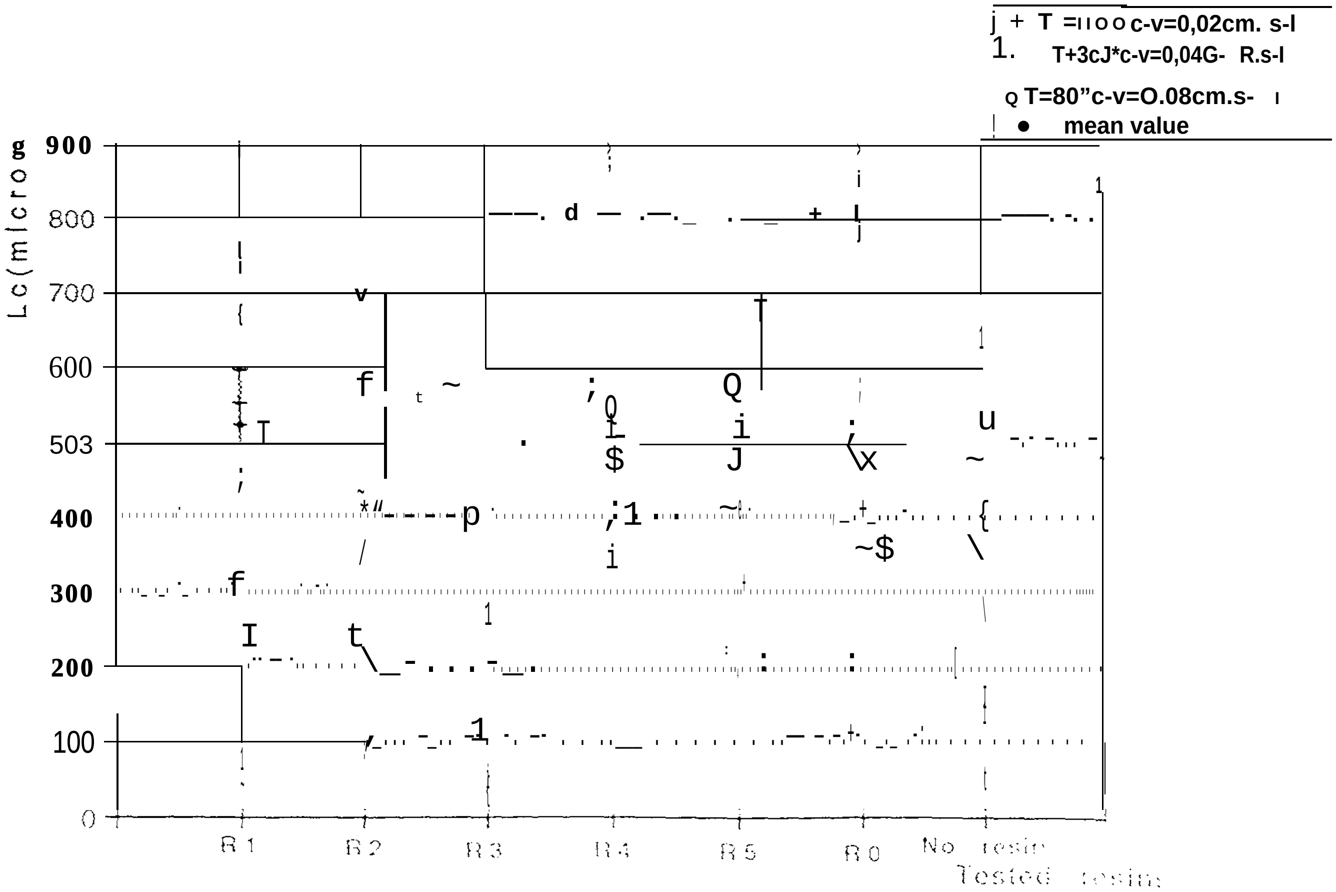
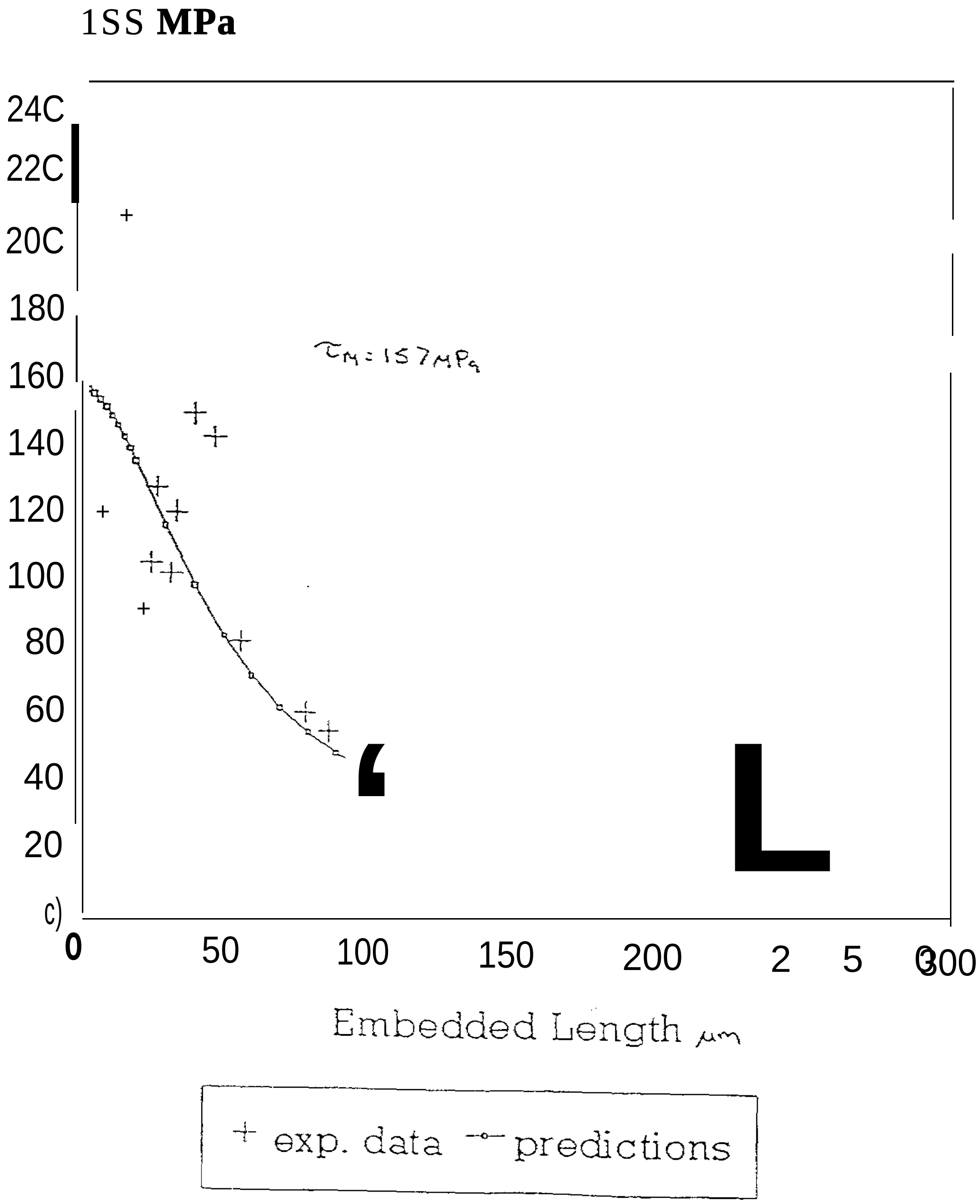


Fig.10 : Tensile tests of embedded T300-508 filaments

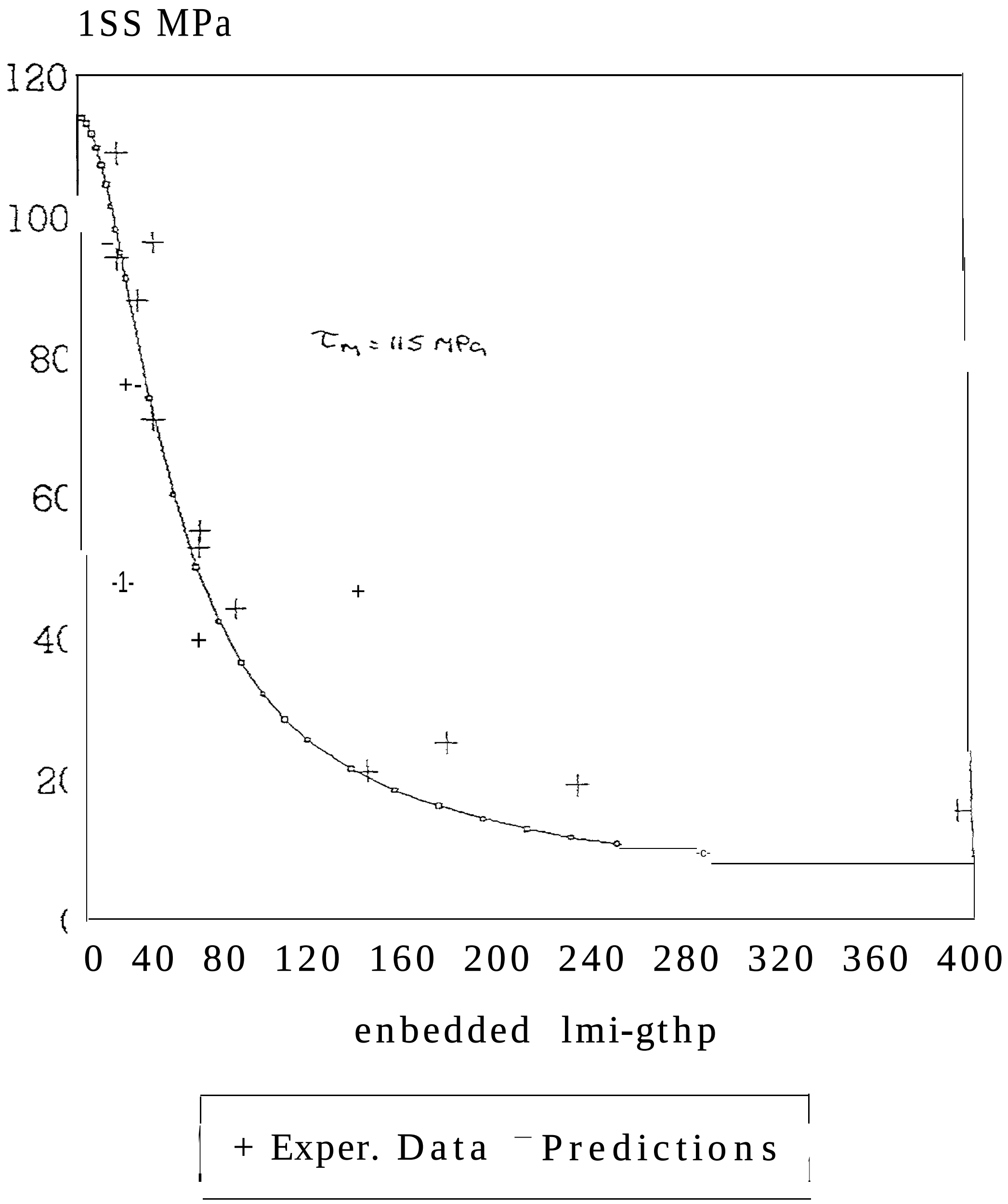
Pull-out Tests

System 1 T (T300/DA508)



Pull-out Tests

System lT' (untreated T300/DA508)



APPENDIX J

SUMMARY OF THE COMPRESSION TESTS PROGRAM ON FLAT LAMINATES (Subtask 1

type of test	clustering of 0° plies	laminate stacking sequence	Loading axis	temperature, no ageing			work sharing							
				RT	90°C	120°C	SNPE	DB	DLR	DRA	NLR	AS	FEUP	IMFL
unnotched compression	1 by 1	(60,-60,60,-60,0,60,-60,0,60,-60,0,60,-60,0,60,-60)s	0°	1T,£,$	1T,£,$	1T,£,$			X					
	2 by 2	(60,-60,60,-60,0,0,60,-60,60,-60,0,0,60,-60,60,-60)s	0°	1T,£,$					X	75				
	4 by 4	(60,-60,60,-60,0,0,0,0,60,-60,60,-60,60,-60,60,-60)s	0°	1T,£,$					X					
	1 by 1	(60,-60,60,-60,0,45,90,-45,0,45,90,-45,0,45,90,-45,0,45,90,-45)s	0°	1T,£,$	1T,£,$	1T,£,$				X				
	2 by 2	(60,-60,60,-60,0,0,45,45,90,90,-45,-45,0,0,45,45,90,90,-45,-45)s	0°	1T						X	55			
	4 by 4	(60,-60,60,-60,0,0,0,0,45,45,45,45,90,90,90,90,-45,-45,-45,-45)s	0°	1T						X				
	1 by 1	(82.5,-37.5,82.5,-37.5,0,45,90,-45,0,45,90,-45,0,45,90,-45,0,45,90,-4	22.5°	1T,£,$				X						
	2 by 2	(82.5,-37.5,82.5,-37.5,0,0,45,45,90,90,-45,-45,0,0,45,45,90,90,-45,-4	22.5°	1T				X	25					
	4 by 4	(82.5,-37.5,82.5,-37.5,0,0,0,0,45,45,45,45,90,90,90,90,-45,-45,-45,-4	22.5°	1T				X						
un. comp. pretension of 0° plies														
	1 by 1	(60,-60,60,-60,0,60,-60,0,60,-60,0,60,-60,0,60,-60)s + pretension	0°	1T								X	RM	
	4 by 4	(60,-60,60,-60,0,0,0,0,60,-60,60,-60,60,-60,60,-60)s + pretension	0°	1T								X		
un. comp in SEM	1 by 1	(0,90,45,-45,45,-45,45,-45,45,-45)s	0°	1T,£,$							X	30		
notched compression d=6mm	1 by 1	(0,45,90,-45,0,45,90,-45,0,45,90,-45,0,45,90,-45)s	0°	1T,£,$									X	
	2 by 2	(0,0,45,45,90,90,-45,-45,0,0,45,45,90,90,-45,-45)s	0°	1T									X	
	4 by 4	(0,0,0,0,45,45,45,45,90,90,90,90,-45,-45,-45,-45)s	0°	1T									X	50
	1 by 1	(0,45,90,-45,0,45,90,-45,0,45,90,-45,0,45,90,-45)s	22.5°	1T,£,$									X	
	2 by 2	(0,0,45,45,90,90,-45,-45,0,0,45,45,90,90,-45,-45)s	22.5°	1T									X	
	4 by 4	(0,0,0,0,45,45,45,45,90,90,90,90,-45,-45,-45,-45)s	22.5°	1T									X	
bolted hole compression d=6mm	1 by 1	(0,45,90,-45,0,45,90,-45,0,45,90,-45,0,45,90,-45)s	0°	1T,£,$	1T,£,$	1T,£,$						X		
	2 by 2	(0,0,45,45,90,90,-45,-45,0,0,45,45,90,90,-45,-45)s	0°	1T,£,$								X	20	
	4 by 4	(0,0,0,0,45,45,45,45,90,90,90,90,-45,-45,-45,-45)s	0°	1T,£,$								X		
	1 by 1	(0,45,90,-45,0,45,90,-45,0,45,90,-45,0,45,90,-45)s	22.5°	1T,£,$								X		
filled hole compression no torque d=6mm	1 by 1	(0,45,90,-45,0,45,90,-45,0,45,90,-45,0,45,90,-45)s	0°	1T,£,$								X		
	2 by 2	(0,0,45,45,90,90,-45,-45,0,0,45,45,90,90,-45,-45)s	0°	1T								X	60	
	4 by 4	(0,0,0,0,45,45,45,45,90,90,90,90,-45,-45,-45,-45)s	0°	1T								X		
	1 by 1	(0,45,90,-45,0,45,90,-45,0,45,90,-45,0,45,90,-45)s	22.5°	1T								X		
notched tension d=6mm	1 by 1	(0,45,90,-45,0,45,90,-45,0,45,90,-45,0,45,90,-45)s	0°	1T,£,$									X	
	2 by 2	(0,0,45,45,90,90,-45,-45,0,0,45,45,90,90,-45,-45)s	0°	1T									X	25
	4 by 4	(0,0,0,0,45,45,45,45,90,90,90,90,-45,-45,-45,-45)s	0°	1T									X	
G2c	UD	(0,0,0,0,0,0,0,0,0,0,0,0,0,0)s	0°	1T,£,$						X	9			
CAI	1 by 1	(0,45,90,-45,0,45,90,-45,0,45,90,-45,0,45,90,-45)s	0°	1T,£,$							X	9		
in situ ultrasonic inspection during comp.		(60,-60,60,-60,0,60,-60,0,60,-60,0,60,-60,0,60,-60)s unnotched, with hole, ..		1T,£,$					X					
initial fibre waviness	1 by 1	(60,-60,60,-60,0,60,-60,0,60,-60,0,60,-60,0,60,-60)s		1T,£,$										
	2 by 2	(60,-60,60,-60,0,0,60,-60,60,-60,0,0,60,-60,60,-60)s		1T										X
	4 by 4	(60,-60,60,-60,0,0,0,0,60,-60,60,-60,60,-60,60,-60)s		1T										X
														X
	1 by 1	(60,-60,60,-60,0,60,-60,0,60,-60,0,60,-60,0,60,-60)s + pretension		1T										X
	4 by 4	(60,-60,60,-60,0,0,0,0,60,-60,60,-60,60,-60,60,-60)s + pretension		1T										X
	1 by 1	(0,45,90,-45,0,45,90,-45,0,45,90,-45,0,45,90,-45)s		1T										
	2 by 2	(0,0,45,45,90,90,-45,-45,0,0,45,45,90,90,-45,-45)s		1T										X
	4 by 4	(0,0,0,0,45,45,45,45,90,90,90,90,-45,-45,-45,-45)s		1T										X
														X
specimens manufacturing				1T,£,$			X							
laminates manuf. + C-SCAN				1T,£,$				X				X		
subtask coordination											X			
specimen manufaruring for UoP											X			

X: 5 specimens per case for compression or tension test
1 case for fibres misalignment evaluation

3 materials:

	program code	table code
DA508/T300	1T	1T
exp resin/T300	2-6T	£
exp resin/T300	2-6T	$

APPENDIX K

DEVELOPED TOOL FOR AUTOCLAVE POLYMERISATION OF A COMPOSITE PLATE UNDER TENSION

On the fig N03, you can see the principle Of a set of tooIs for the polmerisation of ¡plates with different tinsions.

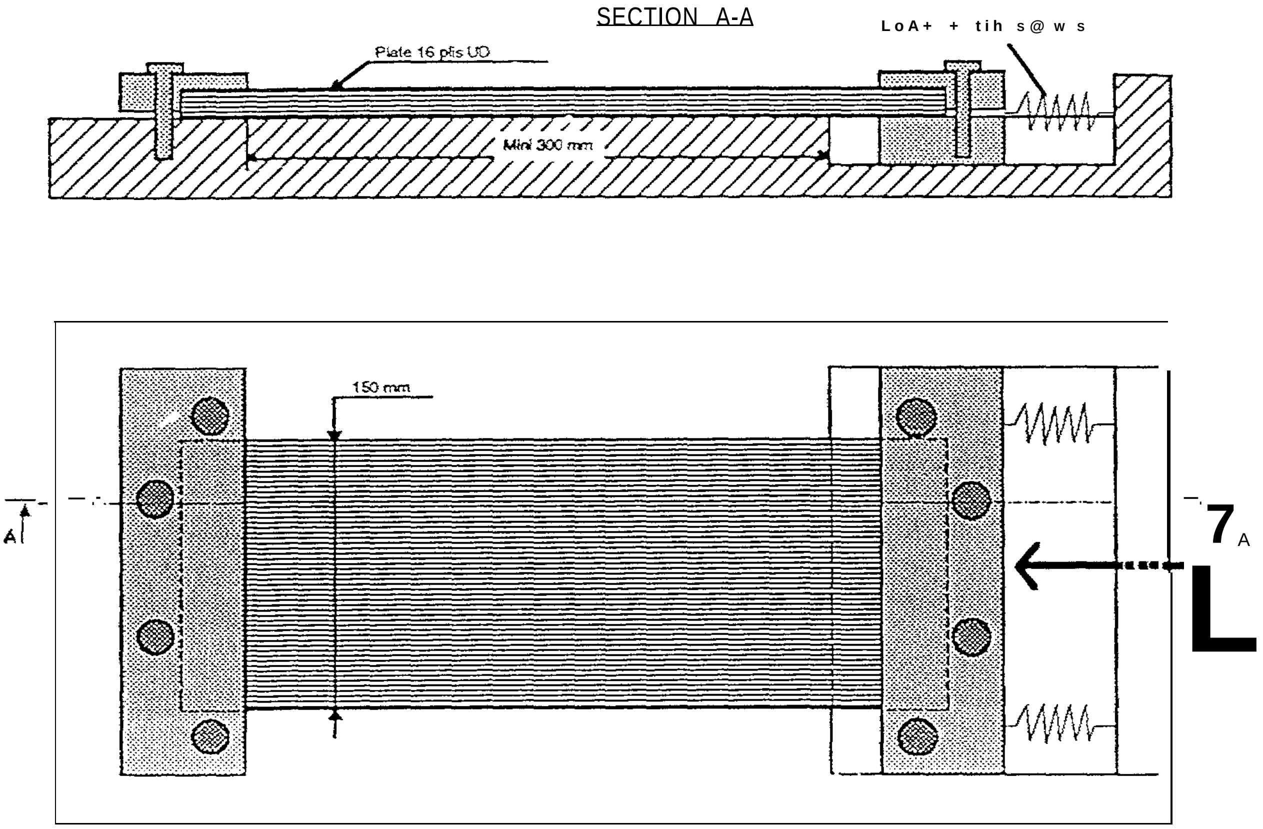


Fig N°3 : *Set of tools for polymerisation of plates.*

‘1’his setting of poIymerisation has the advantage to keep a constant tension Independent, of &e ~emperat,ure (expansion coefficien~) or-r the plate during the polymerisation cycIe

In a first time tJ~e setting allow to apply a tension between O and 400 Kg for a 150 mm. width plate.

The free length of the plate is 300 mm, in order to obtain sound **a!”ea of-** 50 mrn (minimum) where the fibres have a good alignment and where the plate ıas an accurate resin content.

APPENDIX L

PROGRESS IN SUBTASK 1.4 (multi-angles laminates testing)

Status subtask 1.4 programme on 1 July 1994

Partner	Tests	Progress
DB	Unnotched compression under 22.5°	Modified Celanese test rig not ideal. Continue with Airbus test procedure AITM 1.0008 (AS-type) with specimen width 30 mm
DLR	Unnotched compression 3.nsicu US inspection	All specimens manufactured. Start of compression and US inspection 02sE progrfimme
m A	Unnotched compression G~c'ces~{.n~,	No action so far
N m	Specimen manufacturing for Fi3.JP Compression after impact	Manufactured specimens eenc I.'o I?HJP in June 1994 IT specimens ready for testing
AS	Compression of) pre-tension lnmi.l)n!.e~	% d w t i ~ " "-
	Notched compression filled hole torque effect	detailed programme available.
FEUP	Notched compression/tension	17 specimens tested Xrays inspection going on.
IMFL	Waviness measurements	done on received materials

Doc. DGT/DEC/MT N° [illegible]
PR-0003-D/1
October 4th, 1994

FEUP results

Subtask 1A

In this task UP is supposed to carry out tests and damage assessment on notched specimens according to the programme defined in Doc. PE-1401N/3 of May the 1st 1994, in short:

- tension $[(0^\circ)_n/(45^\circ)_n/(90^\circ)_n/(-45^\circ)_n]_{ms}$;

- compression $[(0^\circ)_n/(45^\circ)_n/(90^\circ)_n/(-45^\circ)_n]_{ms}$;

- compression $[(22.5^\circ)_n/(67.5^\circ)_n/(-67.5^\circ)_n/(-22.5^\circ)_n]_{ms}$;

with (m=[illegible] n=1,2 and 4 for 3 different materials (prepregs of different resins).

On the 5th of 1994 plates of the 1T coded material were received at UP's Lab, which were then sent to NLR for preparation of the specimens. These arrived at UP's Lab on the 7th of July 1994. The dimensions of the specimens are:150 [illegible] 30 mm wide, 4 mm thick; and have a center 6 mm diameter hole. 5 specimens/sample were supplied, except the ones cut at 22.5°, for which there were 4 specimens/sample.

Some temion and compression tests were already made at this stage. Tests were conducted at. 0.1 m.rnhnin using as free Ienktihs 60 mm for tension and 30 mm fur compression, the latter according to Aerospatiale’s method. For the tension tests standard wedge action grips are used. For compression a jig based on the CHamxe method was used. Preliminary results obtained which are presented below reseaied I&her conqressiun strengths. As a consequence tabs have to be used to the specimens tested in compression to prevent s~ip al the grips. Two strain gages were glued at the opposite faces to monitor bending deformations i~ the compression tests, wb”ich actually were not obsewed.

At this moment, 1 specimen of each sample has been tested up to breakage, and 2 up to percentages of the tested specimen’s load. These will be X-Ray radiographer and re-tested. The prelimtina~ results are presented in the graphs below. It can be seen that the notched compression strength is higher than notched tension strength. In addi~iun there is a ply chstecing effect, in particular for compression of the 22.5° specimens.

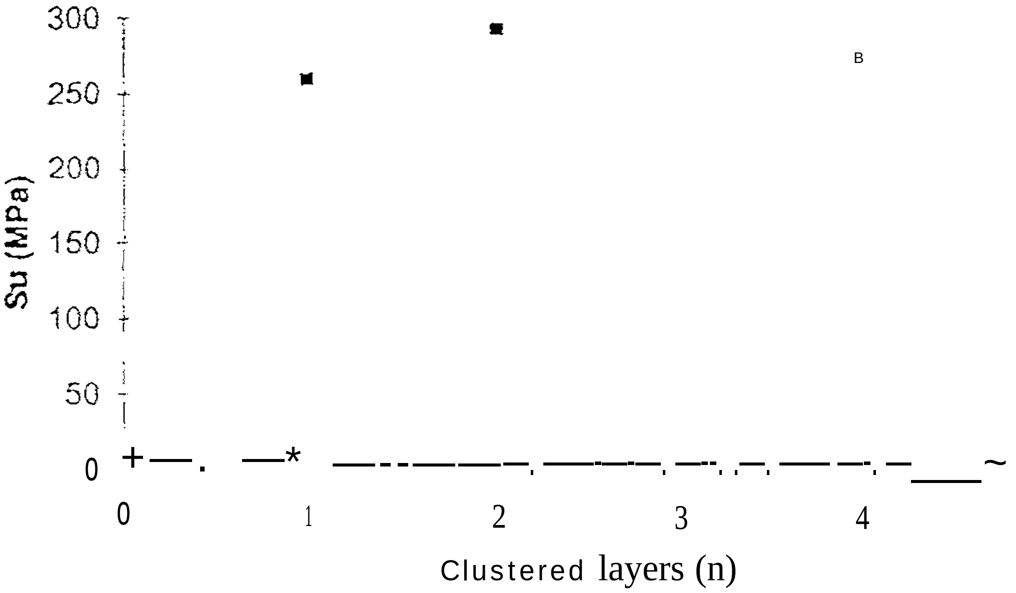


Fig. 1: Open-hole tension results for the $[(0^\circ)_n/(45^\circ)_n/(90^\circ)_n/(-45^\circ)_n]_{ms}$ specimens, IT material.

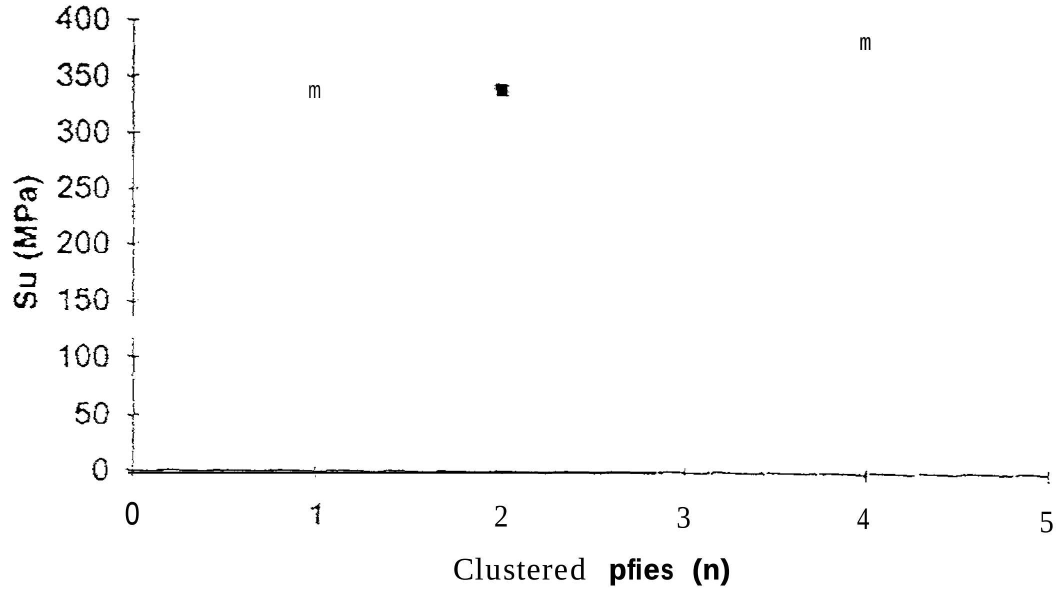


Fig. 2: Open-hok compression results for the [(0°~i(45*)n/<900)n{(-45 °J.Jm specimens, 1 T mtexial.

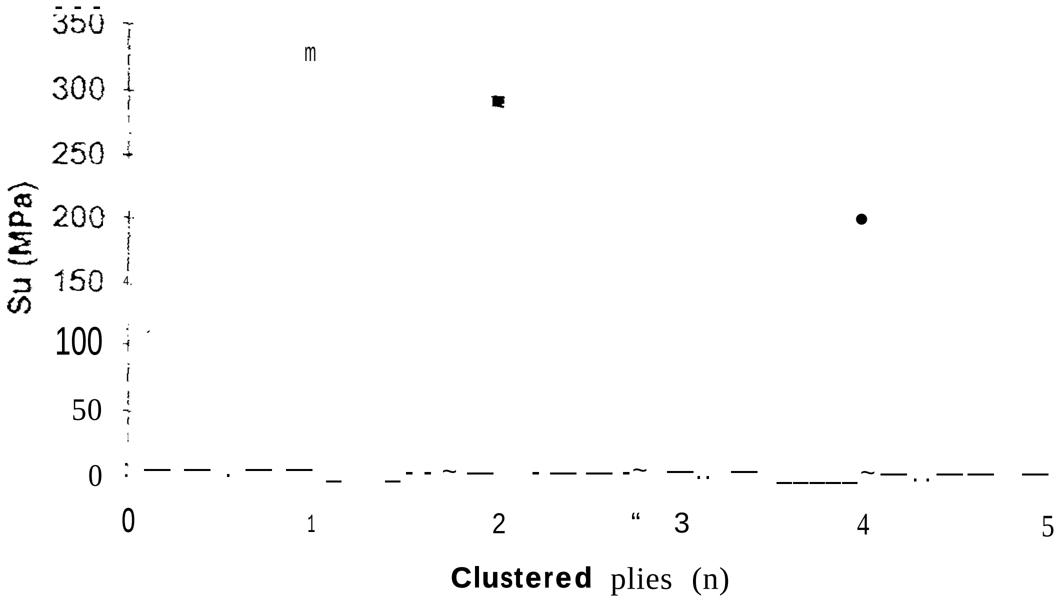


Fig. 3: Open-hole compression results for the $[(22.5^\circ)_n/(67.5^\circ)_n/(-67.5^\circ)_n/(-22.5^\circ)_n]_{ms}$ specimens, IT material.

APPENDIX M

DEVELOPMENT OF THE SIMULATION TOOL (subtask 1.5)

1- INTRODUCTION

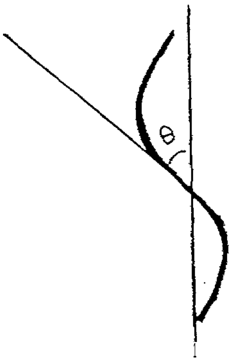
Our purpose is to focus on failure mechanisms of unidirectiormal composite unde compression. Finite elements code ELHNI and CATIA meshing assistance permit a realistic composit modelisation. Our study is based on the modelisation of an unit ceH which represents one fiber embedde with matrix.

Simulations show us which are the parameters governing the failure of our cell : lengtł misaligneinent, matix modulus, elastic limit of the matrix,...

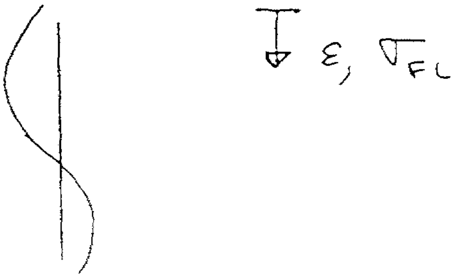
The first step consists in trying to establish the influence of the difierent parameters with a 2I cell; secondly a 313 cel~ allows us to predict the strength in compression for new materials.

2- MODELISATION OF A 2D CELL

The misalignment is simulated by a fiber which has a sinusoid wave geometry, characteriz by its length and ik maximum angle 8 (fig. 1).



The failure of lhe cell is deter-miiled by the evolution of OFL (the buckling stress) versus (global strain imposed to oui- cell).



The main hypothesis is that local geometrical defects create overstresses which lead the cell t ftilure. To evaluate the influence of the matrix modulus EM, two simulations were performed with values of EM: 3700 MPa and 6000 MPa. The first hypothesis is that the matrix is elastic : in that case, th variation of the buckling stress GFL is very important in regard of the variation of EM. But such behaviour is not realistic for the matrix : it is establish that epoxy matrix have an elasto-plastic behaviou (see subtask 1.3). In that case, the figure 2 shows us that for a variation of EM of 60%, the buckling stress CSFL doesn’t vary more than 10“/O { oo is the elastic limit of the matrix).

The importance of the maximum local misalignment 8 can be evaluated in relatioti with ~. The figure 3 illustrates the dependency of GFL on 8; the 3 curves are calculated for 3 values of ~0.(50 100 and 150 MPa). These simulations ar~ made on 3 cells characterized by local misalignment 0: the values are 0.45°, 0.9° and 1.8°.

Then we have tried to take into account the experimental behaviour of the fiber. As ~v can see on the figure 4, fibers have a non-linear behaviour in compression : the modulus of T300 fibe decreases when the stress increases in compression. With this more complex modelisation of our cell FL decreases of about 10%, The explanation of the bound between GF~ and the matrix is given by th figure 5 which illustrates the shear stress ayz in the matrix along the fiber. The overstresses leads t ftilure when the elastic limit GO is reached.

3- MODELISATION OF A 3D CELL.

The cell is simulated by a central fiber embedded with matrix and surrounded by 4 quarters fiber to assure the repetitivity of the cell.

The fiber has a sinusoid wave geometry in the plane XY only , The misalignment in dl plane XZ is much lower Ihan tha~ in plane X2, so it is negligible.

The evacuation of’ the influence of the different geometrical and mechanica~ parameters i made in the same way than for the 2D cell.

The figure 6 shows us the evolution of GF~ versus 8 with 2 different values for the matri modulus. The curves with a linear behaviour of the matrix show us a great influence of’ E.h~, but this is n realistic because the matrix has a non-linear behaviour. In that case, the variation of E~~has lo~ consequence on cqqj.

The figure 7 shows us the evo]u[ion of G~L versus 6 for different vaiues of cr~ (the elasti limit of the matrix). With such a set of curves, we are able to predict, numerically, tile stress of failure f a U D composite, having some geometrical and rnechanicai parameters.

We must precise that a simulation on a 3D cell with a non-linear behaviour of the nl~trix and non-linear fiber costs about 2 hours CPU (with a IBM ES9000).

The di fferencc bet~vcen 2D simulations and 3D ones is shown in the figure 9. l-he matrix the ~i> ce[i has only one shear stress u~y., but the 3[] cc]} matrix is under the influence Of z s[lcar Slrcssc n~y and ax~ : (he COlllbi[ld ill flucllce of the Lwo stresses give di ffer’enl \:i]ucs ibrl!ic I)[ic!i]ing s(PL’s of the W[l.

4- CC)h’CLUSIONS

We have built a numerical tool, based on a 3D cell. With that tool, we are able to predict tl failure of a I.JD composite in compression. Some geometrical and mechanical parameters have to l determined : the local misalignment, the elastic limit of the matrix,...

We must precise that, as our ceH is just a representation of the reality, the value of tl misalignment 8 is not necessary the mean value measured by the IMFL. Our modelisation involves tl establishment of a relationship between the measured misalignment and the value used in the cell.

The next step will be to test the validity of our modelisation with the materials studied in ol program.

/Fig. 1:20 cell: sinusoid fiber and matrix

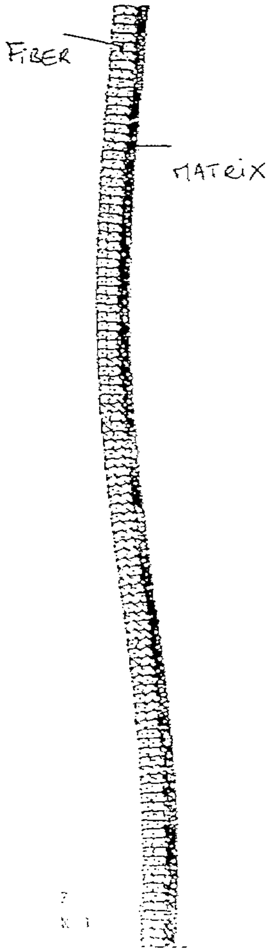


Fig. 2 : Variation of the buckling stress - 2 cases : elastic and elastoplastic matrix with 2 values of Em

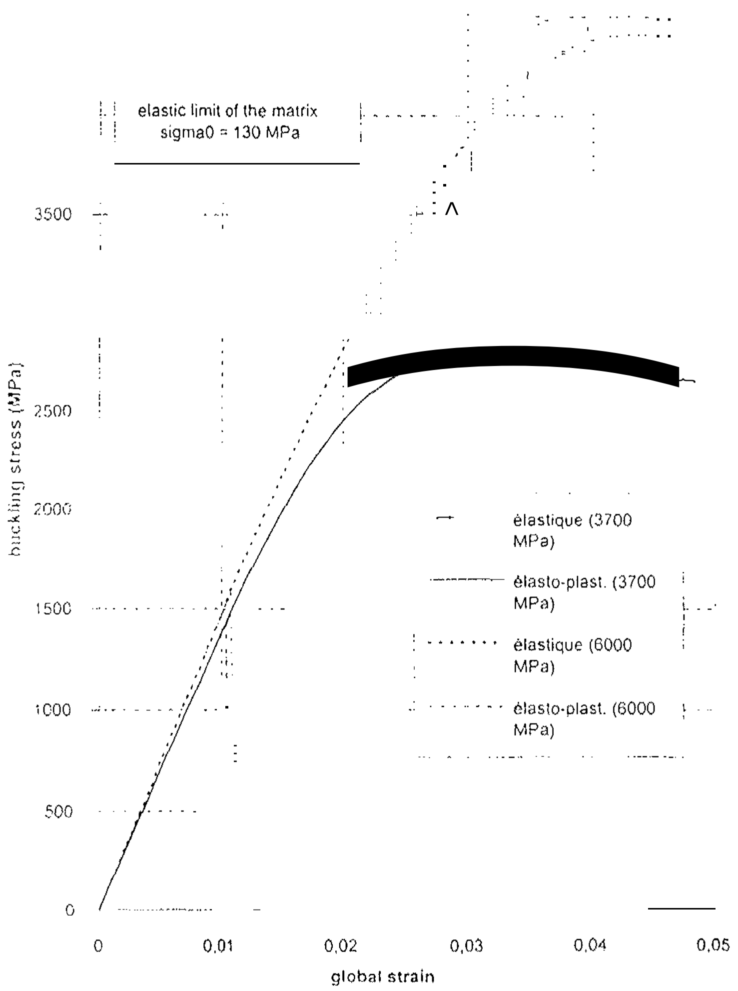
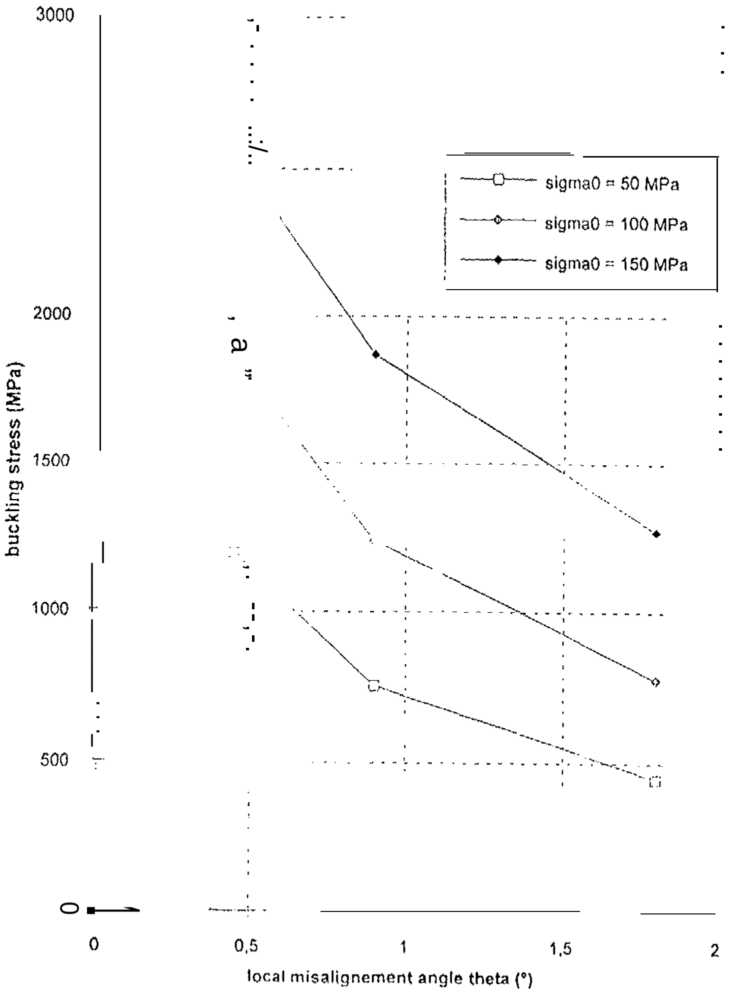


Fig. 3 : Influence of the misalignement in the 2D cell for different values of the elastic limit of the matrix



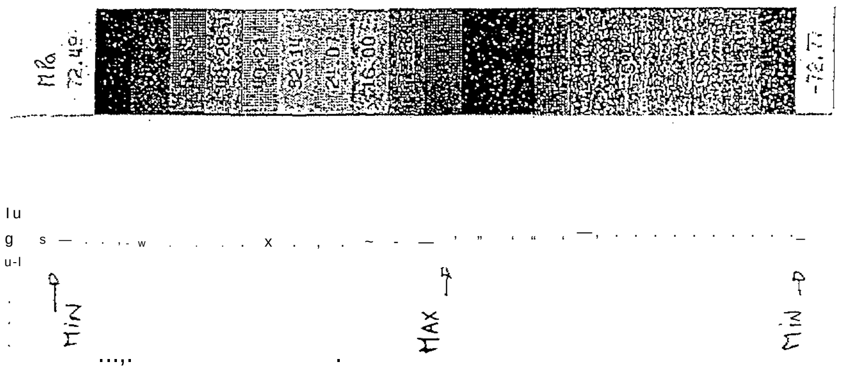


Fig. 5 : variation of the matrix shear yz in the 2D cell

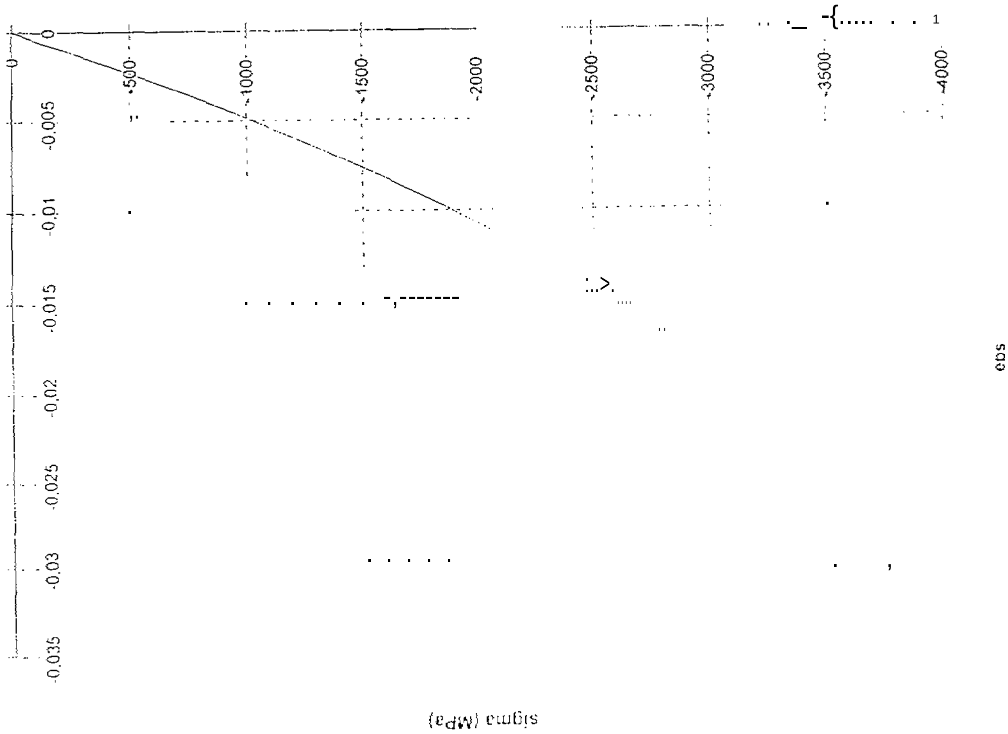


Fig. 4 : Behaviour of T300 fibers in compression

Fig. 6 : Influence of the matrix modulus Em in the 3D cell

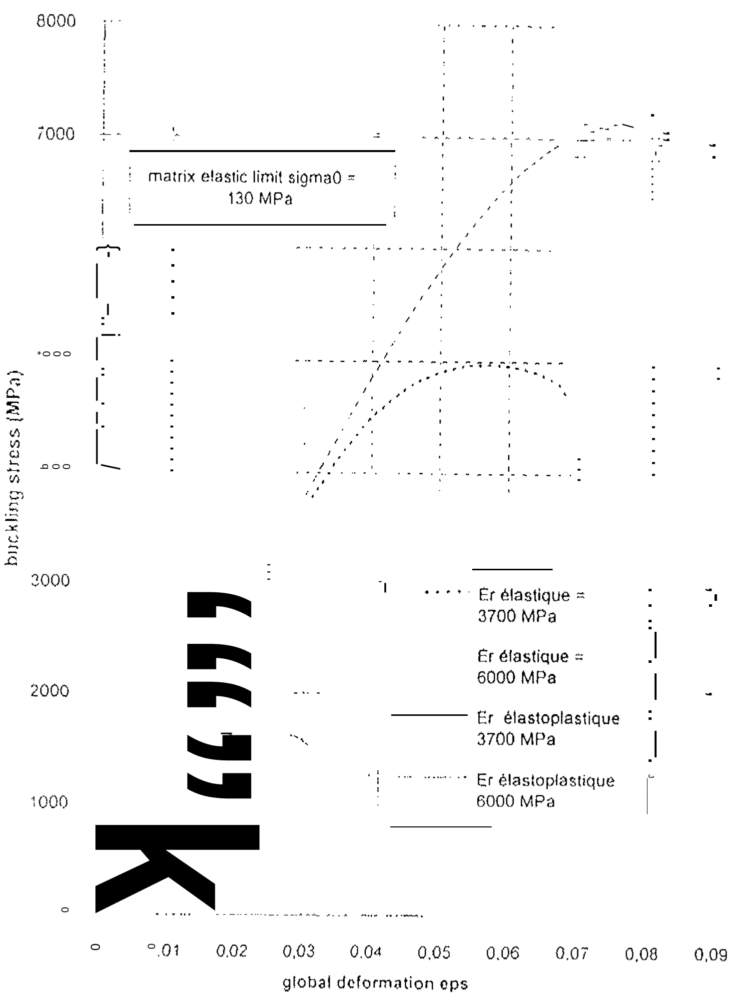
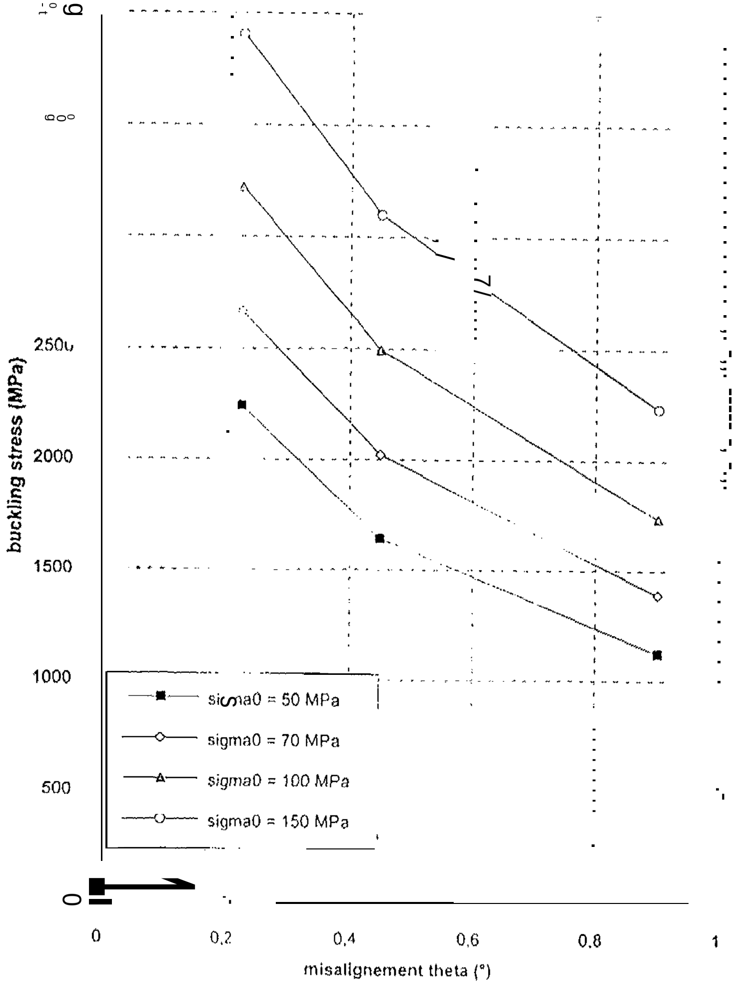


Fig. 7 : Influence of the misalignement theta in 3D cell for different values of the matrix elastic limit



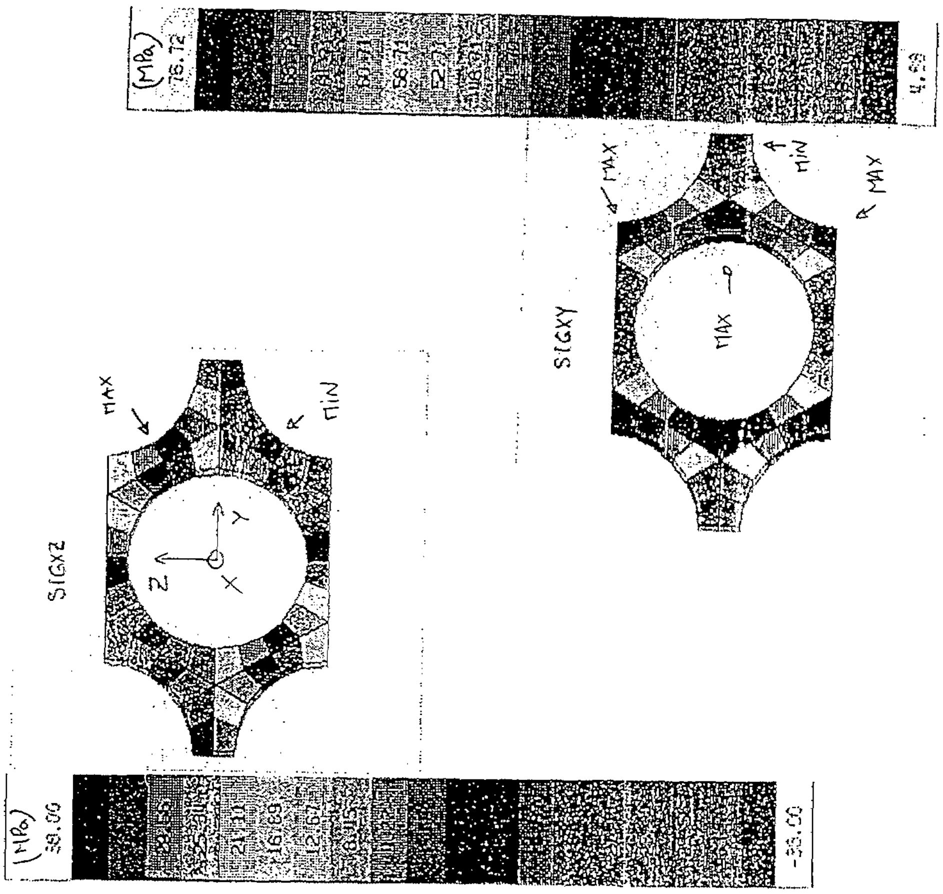


Fig. 9 : variation of the matrix [illegible] cell

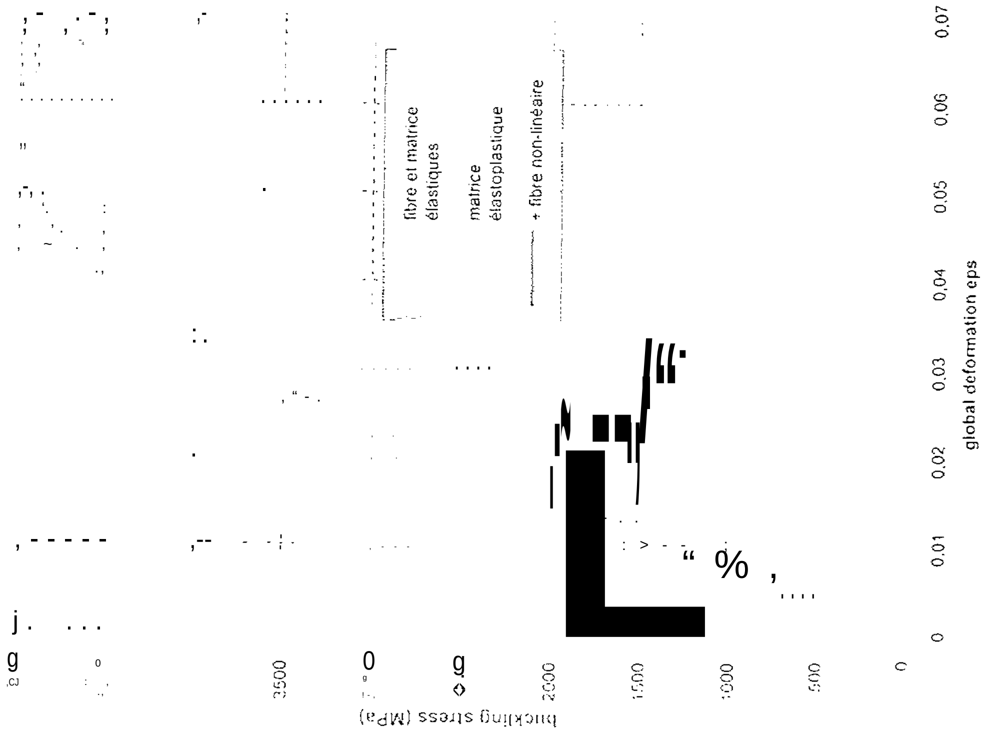


Fig. 8 : Influence of all the non-linearities with the 3D cell

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PR-0003-D/l
October 4th, 1994

APPEN^T DIX h'

DEI.JVEWU3LES PRESENTATION FOR TASK 1 COMPLETION

Document code	resuo*nsibie* partner	content
DE- 100 I -DKI OveraH task 1 synthesis	Dassault	Conclusions of results analysis, simulation tool.
DE-1 101-EMO subtask 1.1	DassauIt	
DE-1201-MI subtask 1.2	DRA	Working group leader, synthesis of results analysis within each subtask
DE-1301-0/O subtask 1.3	O ~ E ~	
DE-140 I-N/() subtask 1.4	NLR	
DE-1 501-D/O subta.sk 1.5	Dassault	
NT-1 . ..-./0	all	Technical report from each partner, detailed results from each subtask contribution.