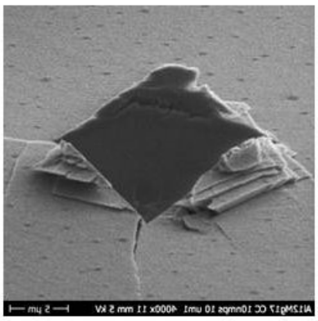
Summary report

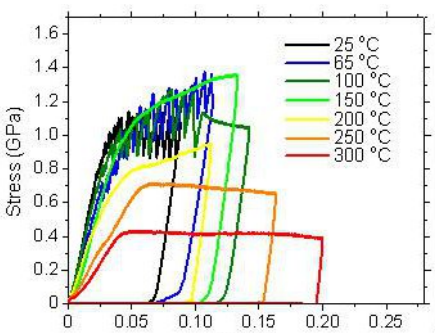
The plastic deformation of materials, such as metals, can be easily studied because they are ductile. Plastic flow also occurs in brittle materials and is critical in determining their in-service resistance to both mechanical and electrical loading. For instance, it can determine the lifetime either of a hard coating on a cutting tool, or of an actuating device. However, it is much more difficult to study plastic flow, because the sample simply breaks.

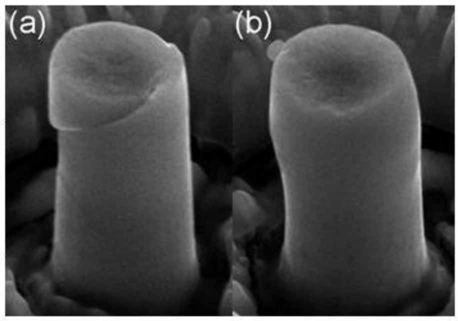
One way of stopping cracking is to apply a large pressure, or, more easily, by indentation. However, it is known that very high confining pressures can modify the atomic structure around defects that cause plastic flow, which is directly associated with the resistance to flow in hard materials. Indentation, which relies on constraint from the surrounding material, has the further disadvantage that, in more complex crystal structures, slip must occur on different crystal planes and directions. These different systems often have very different critical resolved shear stresses. A hardness measurement depends mainly on flow in the hardest system, so interrogating soft slip systems by indentation is not possible, although it is often flow on these weaker systems that causes failure.

A simple way of studying plastic flow in brittle materials is urgently required.

It has been known for some time that reductions in scale, to sample sizes of the order of a micron or so, can suppress fracture, although the exact reasons are still a matter of debate. The aim of this project is to explore the deformation behaviour of brittle materials using micron-sized samples, known as micropillar compression, and to compare the results obtained with more commonly used methods such as indentation, over a range of temperatures temperatures. Work has focused on two complex metallic alloys: $Mg_{17}Al_{12}$, which is cubic and the orthorhombic (o) form of $Al_{13}Co_4$.







$Mg_{17}Al_{12}$, with a melting point of approximately 460 ºC, was studied from 25 ºC to 300 ºC. The hardness was determined by nanoindentation showing a pronounced drop at temperatures above 150 ºC. The flow stress was evaluated from micropillar compression experiments and showed the same trend. Both, nanoindentation and micropillar compression showed that serrated flow was taking place at lower temperatures. The size of the serration was dependent on the strain-rate whereas the hardness was not. Because of the similarity with the behaviour of a metallic glass, the possibility that yielding was influenced by a normal stress component was investigated using finite element simulations. However, the pile-up pattern surrounding the residual indents suggest that, unlike in metallic glasses, the crystallography plays an important role in the deformation of this material.

The single crystal deformation behaviour of o-$Al_{13}Co_4$ has been studied using indentation, microcompression and transmission electron microscopy, from 25 ºC to 600 °C. While most materials lose their hardness with increasing temperature, anomalous yielding behaviour was observed for o-$Al_{13}Co_4$ above 226 °C, where the 2% flow stress increased from approximately 2 GPa to almost 4 GPa by 600 °C. This was the first time that anomalous yield had been observed in a complex metallic alloy. Plastic deformation occurred most easily along the (001)[010] slip system, as observed at higher temperatures. However, as the temperature increased, the deformation pattern changed to one consisting of defects running perpendicular to the loading axis. Serrated flow was also observed at all temperatures except 600 °C, where flow was mainly homogeneous.

The techniques developed here have general applicability and can be used to explore plasticity in brittle materials in a range of systems.

The usefulness of brittle materials is often limited by their ability to deform at low and intermediate temperatures. This work is the first study of this type in a new and emerging class of materials. These materials have potential industrial applications in tribology and surface protection, both mechanical and chemical. For instance, very low friction coefficients have been measured when metal-matrix/CMA composites slide against hardened steel in ambient air. Other areas of interest include hydrogen storage, as these materials contain the icosahedral structure so common in Laves phases, as well as thermoelectric, catalytic and magnetic applications.

The work here has exploited a European world lead in the ability to carry out small-scale tests over a range of temperature. Exploiting these techniques, it has been shown, for the first time, that these materials do deform by dislocation motion below the ductile-brittle transition temperature. Despite this, their overall behaviour has many features in common with a metallic glass. Basic measurements of stiffness, flow stress, hardness and toughness have been made. These are materials with high flow stresses as a fraction of their stiffness and they are brittle. There is evidence, as in many intermetallics, that the grain boundaries are particularly so, suggesting that gettering elements or compounds may be required, such as B used to 'ductilize' Ni_3Al.

$Mg_{17}Al_{12}$ shows serrated flow and a decreasing flow stress and hardness with increasing temperature. $Al_{13}Co_4$ also shows serrated flow, but the flow stress increases with temperature, *anomalous yielding*. This is the first time this has phenomenon has been observed in a complex metallic alloy. It has been suggested that this might arise due to the nature of dislocation in these materials, suggesting that this might be a much more widespread phenomenon than so far generally assumed.

Dissemination is taking place through journal papers. Three are to be submitted. The first is on $Al_{13}Co_4$, the second on $Mg_{17}Al_{12}$ and a third on the study of deformation in $Al_{13}Co_4$ using in-situ Laue X-ray diffraction. However, this work has also attracted the interest of the CMA-community, notably the C-MAC network. There have been other (invited) conference presentations including the ESMC meeting in Graz in 2012, and ICMCTF in San Diego, 2012, as well as the Nanomechanics meeting in Portugal in October 2013. There was also a very successful MRS meeting at which the results on $Al_{13}Co_4$ were first presented. This led to a developing contact with Drs Feuerbacher and Heggen. Activities are also continuing with Dr J. Michler at EMPA, CH on in situ, high temperature testing, with Prof. Dr Sandra Korte at FAU Erlangen-Nuremberg, also on high temperature testing, and with Dr F. Giuliani on Laue diffraction, where the size of the unit cells imposes particular difficulties with the analysis.