ESRF	**Experiment title: Mechanical hysteresis in layered crystal structures**	**Experiment number**: MA988
Beamline: ID-15B	**Date of experiment**: from: 25/06/2010 to: 29/06/2010	**Date of report**:
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Report:

MAX phases, with the general formula $M_{n+1}AX_n$, where n = 1 to 3, are transition metal carbides and nitrides that show a significant hysteresis effect in their loading-unloading curves. Existing theories attribute this hysteresis to incipient kink bands whose spacing is sufficiently small that they recombine on unloading. An alternative explanation for the hysteresis is that the anisotropy of flow allows some grains to plastically deform, while others remain elastic, causing intergranular stress accumulation. The aim of this work was to examine the idea whether the hysteresis that is observed in these materials might be associated with the onset of plastic flow in some grains, accommodated by elastic deformation in others, rather than kinking.

Test specimens of cylindrical cross-section 3 mm in diameter and 7 mm long were cut from blocks of hot-pressed Ti_2AlC and Ti_3SiC_2 by electrical discharge machining abd compressed at room temperature, 700 °C and 1,000 °C using an electro thermal mechanical test (ETMT) rig. X-ray diffraction measurements were carried out on beamline ID15-B. The current was adjusted manually as the sample was deformed to maintain a constant temperature which was monitored with a pyrometer. Intergranular elastic strains were determined from the diffraction data.

Some difficulties were experienced with the measurements of load/displacement, particularly at higher temperatures. As might be expected the best results demand that displacements be measured on the sample. However there was also drift of the maximum measured load by a significant amount. The origin of this is not clear. To minimise any complications, analysis of the results obtained was started by determining the intergranular strains for each peak at zero load following each cycle. It has been found that that intergranular strains developed during the first loading cycle and remained approximately constant after unloading in the subsequent cycles. Both positive and negative intergranular strains were measured, as would be expected. A large negative intergranular strain is seen to develop in grains where the reflections were obtained from the (002) planes, indicating slip did not occur in these grains. However positive intergranular stresses were obtained in those grains where diffraction data was obtained from the (00.12) and (00.14) planes, indicating yield had occurred in these grains. It is clearly seen that yield is occurring in those grains whose basal planes lie at an inclined angle to the loading axis, i.e. not perpendicular or transverse to the loading axis. However if some grains were to deform by the formation of incipient kink bands then these too would give rise to internal stresses.

However incipient kink bands would be expected to completely relax at zero load, so that there would be no residual intergranular strain at zero load. The observation that the intergranular strains, at zero load, increase rapidly during the first cycle and then only increase more slowly is also support for the idea that the hysteresis is associated with mechanism other than incipient kink bands. support the idea that this it is caused by the build up of intergranular strains caused, predominantly at least, by yielding in some grains and not in others. The hysteresis in load/unload curve is associated therefore with plastic flow occurring in one direction and then being driven backwards by the internal stresses. The extremity of the plastic anisotropy in these materials is consistent with the observation that the strain can be almost completely recovered [1].

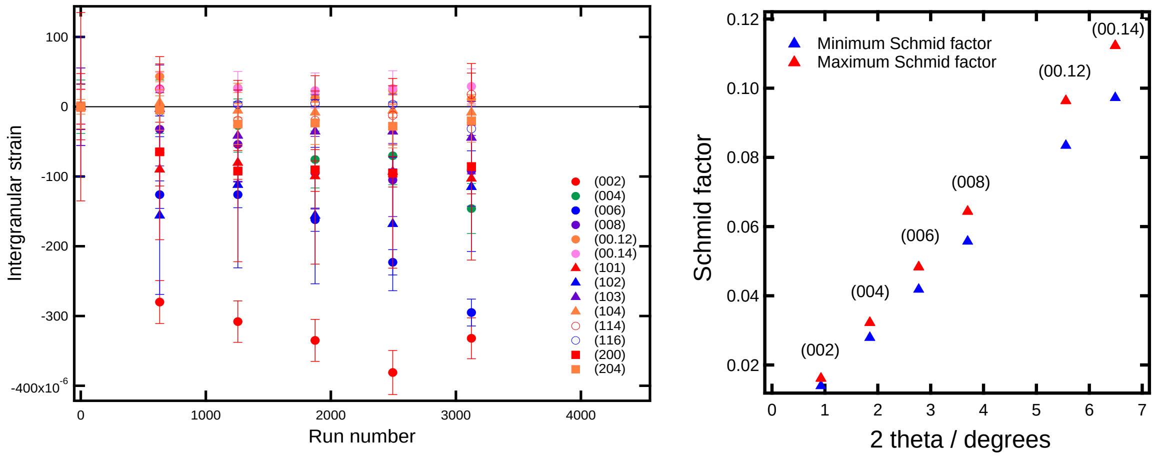

Figure 1 (a) The intergranular strains estimated from the diffraction data after different numbers of cycles and (b) the values of the Schmid factor predicted on differenmt planes.

It can be seen from figure 1a that the (004), (006) and (008) basal planes show lower values of negative intergranular strain than those determined from the (002) planes. The value of 2θ for the (002) planes is very small and 2θ increases with an increasing value of *l* for the (00l) basal planes. For the (002) planes, the Schmid factor is very small and there is very little critical resolved shear stress on the (002) plane, so it is unlikely that deformation will occur on these planes. As the value of *l* increases, and with it a decreasing 2θ, the Schmid factor increases, figure 1b. However, there is not a systematic reduction in the amount of negative intergranular strain seen on the planes – (006) is between (002) and (004). This could mean that another factor, such as texture in the sample or elastic anisotropy is having an effect and these are being investigated.

Despite some experimental difficulties, the experiment has clearly demonstrated that:

(i) intergranular strains build up during deformation;

(ii) that they are retained in the sample even when fully unloaded

These observations are inconsistent with hysteresis being associated with incipient kink bands, and support the idea that this it is caused by the build up of intergranular strains caused, predominantly at least, by plastic anisotropy leading to yielding in some grains and not in others. The hysteresis in load/unload curve is associated therefore with plastic flow occurring in one direction and then being driven backwards by the internal stresses.

References

1. M.W. Barsoum, T. Zhen, S.R. Kalidindi, M. Radovic, and A. Murugaiah, "Fully reversible, dislocation-based compressive deformation of Ti_3SiC_2 to 1 GPa", *Nature Materials*, **2** (2003) 107-111