



Experiment title: Study of Dynamic precipitation in an Al-Zn-Mg alloy by means of in-situ Small Angle X-ray Scattering in deformation

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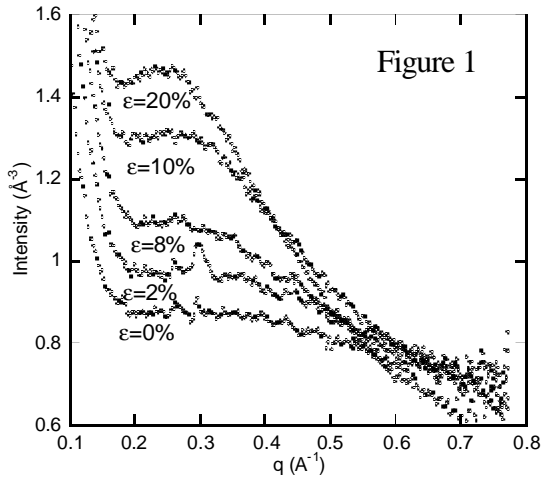
Report:

The materials studied in this experiment are based on the Al-Zn-Mg alloy system. These alloys are of great industrial importance since they are used for their high specific mechanical strength in a wide range of transportation applications, especially in the aerospace area. This mechanical strength is gained through fine scale precipitation of intermetallic particles in a volume fraction of 2 to 5 vol%, in a size range of 1 to 10 nm. It is of course fundamental to gain a good understanding of the precipitation behaviour of these alloys in the various steps of their process history in order to optimise their end use properties.

Among the various steps encountered in their thermomechanical process, several involve plastic deformation in a supersaturated state (i.e. an unstable solid solution in the two-phase region of the phase diagram). This plastic deformation may modify the precipitation behaviour in several ways, which always involve an acceleration of the kinetics as compared to static precipitation (i.e. precipitation in the absence of plastic deformation) : role of dislocations on precipitation nucleation (heterogeneous precipitation), role of dislocations in terms of pipe-diffusion of solute, elastic interactions between dislocations and solute atoms, role of dislocations in terms of "shuffling" atoms. Some preliminary experiments carried out at ESRF in 1997 [1] showed that dynamic precipitation could occur significantly at low temperatures (ambient), at rates several orders of magnitude higher than static precipitation. However, measurements were performed ex-situ, which limited in a large extent the precision of the data, and the number of experimental situations which could be investigated.

The aim of the present experiment was to investigate precipitation during plastic flow in-situ, using a tensile apparatus developed by Laurent David and co-workers of INSA Lyon (GEMPPM). Several alloys were investigated, as well as several initial states : full solid solution (maximum supersaturation), or containing some Guinier-Preston zones (after some ageing at room temperature). Most experiments were carried out at room temperature, except a few which were performed at 100°C.

In this report we will present some of the most representative results, on the room-temperature deformation of fully solutionized alloy AA7010 (Al-Zn-Mg-Cu).



In figure 1 we can see the SAXS evolution during the deformation of the material in-situ under the beam (in transmission). It appears clearly that precipitation is occurring, at a fast rate, since the deformation takes place in a few minutes. A more quantitative analysis is shown in Figure 2 and 3, which show the evolution during the tensile test of the particle radius and volume fraction as calculated from the SAXS spectra, compared to what happens during static precipitation. Clearly plastic deformation helps the precipitation, most remarkably in terms of volume fraction, but also in terms of radius, after some sort of incubation period.

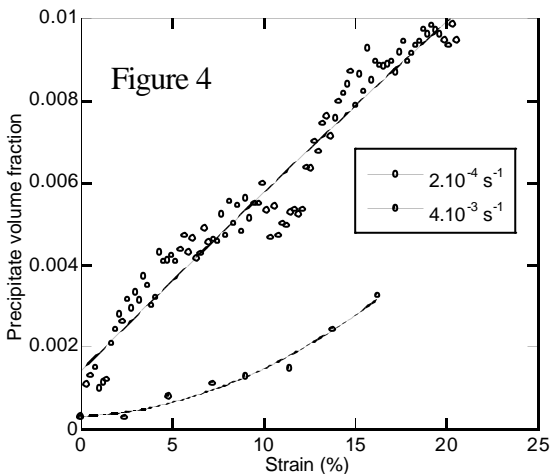
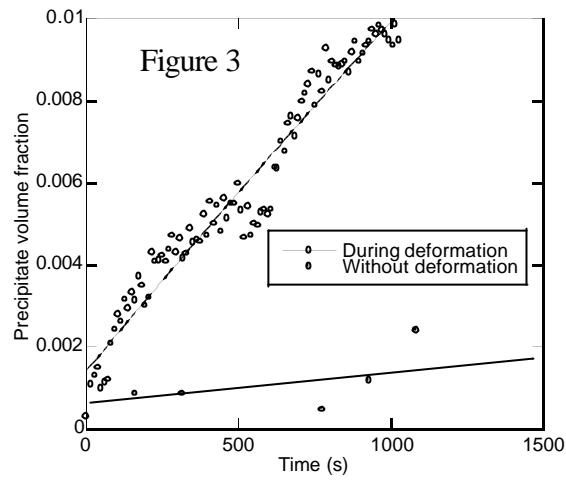
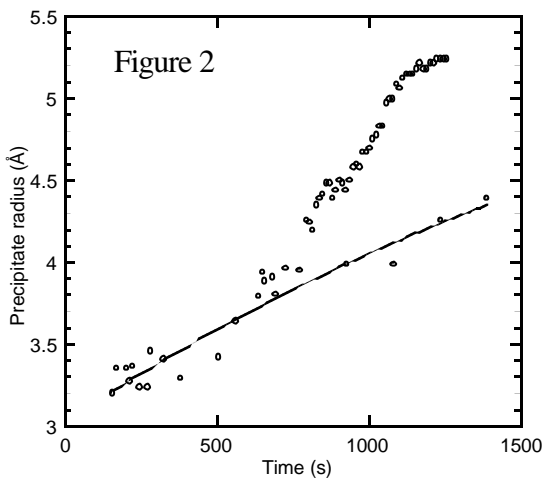


Figure 4 illustrates the effect of one deformation parameter on the dynamic precipitation phenomenon, namely strain rate. When the strain rate is increased by a factor of 20, dynamic precipitation occurs in a much smaller extent. This observation gives some hints about the mechanism of dynamic precipitation : one likely mechanism is solute migration towards the dislocations when they are stopped at obstacles (such as particles, grain boundaries or other dislocations), during the so-called waiting time t_w . It is well known that there is a direct relation between this t_w and the strain rate.

Conclusions

In conclusion, it is the first time that low temperature dynamic precipitation is observed in-situ, and we have been able to determine quantitatively its characteristics, such as evolution of the size and volume fraction of the precipitates created, and influence of strain rate. Publication of these results is under way.