In-Situ Study of Precipitation Kinetics during Thermomechanical Treatments in a 7xxx Series Aluminium Alloy by Small Angle X-ray Scattering

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Aims of the experiment and scientific background

The precipitation processes and kinetics during isothermal and non-isothermal heat treatments of 7000 series aluminium alloys have been thoroughly investigated [1, 2, 3]. However, there are a number of practical situations where precipitates form along with an imposed stress or strain. For instance, age forming or creep age-forming are processes used in the aerospace industry to manufacture complex-shaped panel components, allowing particularly low residual stress levels [4, 5]. In these processes, part or all of the heat treatment is carried out simultaneously to a forming operation, which results in a plastic strain heterogeneously distributed in the material. Such an imposed strain and stress can have a large influence on the kinetics of the reaction, the nature of the phases formed and the resulting mechanical properties [6] ; yet it has received very little attention in the literature, and the extent of the coupling between precipitation kinetics and imposed stress and plastic strain are largely unknown.

SAXS (Small-Angle X-Ray Scattering) is an appropriate experimental tool to study precipitation, allowing a statistical description of the main precipitates parameters, such as the precipitate mean size, density and volume fraction [2, 7]. While it is quite common to carry out in-situ measurements of precipitation kinetics during heat treatments [8, 9, 10], performing a similar task in thermomechanical (TMT) conditions is very seldom. We developed a micro-tensile machine with a built-in furnace, allowing to carry out in-situ SAXS measurements in TMT conditions. This tool has been used in conjunction with a synchrotron X-ray source to study the coupling between stress / strain and microstructural evolution during TMT heat treatments representative of age forming operations, i.e. tensile tests followed by relaxation at imposed displacement, and creep tests, on the AA7449 aluminium alloy. We have varied the parameters of the TMT treatment (strain rate, imposed plastic strain, imposed load, …). Both the stress / strain parameters and the microstructural parameters (size, volume fraction, precipitate density) have been followed continuously during the TMT treatments.

Experimental

Tensile device

The tensile device is actuated by a step motor, located outside the vacuum chamber. A large hole in the back allows the SAXS signal to be measured after transmission through the sample. The load is measured by a piezoelectric sensor and the displacement by an inductive sensor. The sample is heated by conduction through the two grips, on which resistive cables are welded. Temperature control is performed by thermocouple measurements in the grips. The temperature range is between room temperature and 400°C; the load can reach 1kN and the displacement 6 mm, corresponding to 150% strain.

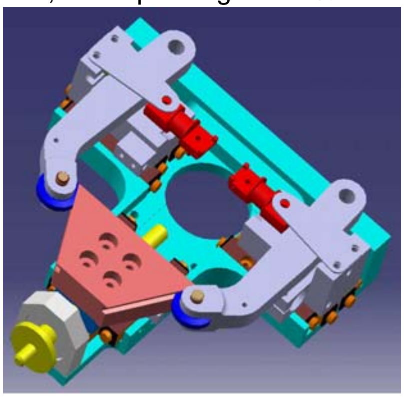

Figure 1. CAD image of the tensile device

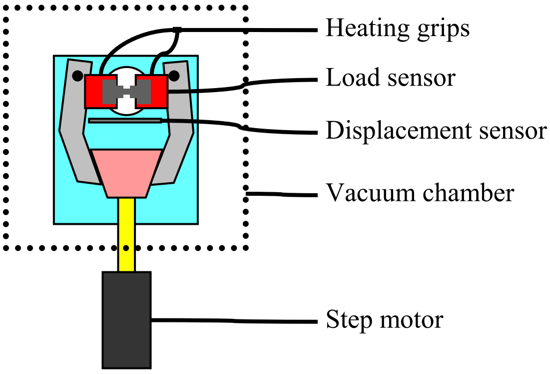

Figure 2. Scheme of the tensile device

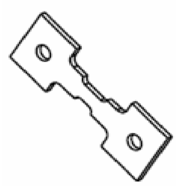

Figure 3. Tensile sample

Material and TMT treatments

The studied material is a 76 mm thick plate of AA7449 alloy, a wrought aluminium of the Al-Zn-Mg-(Cu) family, which composition in the main alloying elements is about 8.1% Zinc, 2.3% Magnesium and 1.8% Copper (all in wt%).

The thermomechanical (TMT) treatments (see black line on figure 4) were carried out on samples subjected to a partial heat treatment (pHT), consisting of a solution heat treatment, a quench in cold water, and the same ageing treatments as compared to the T7651 except that no pre-stretch was applied and that the heat treatment was interrupted (water quench) at various ageing times.

30 minutes long TMT were performed while measuring the precipitation evolution: creep tests (various temperatures and stresses) and tensile tests (various strain rates and strains)

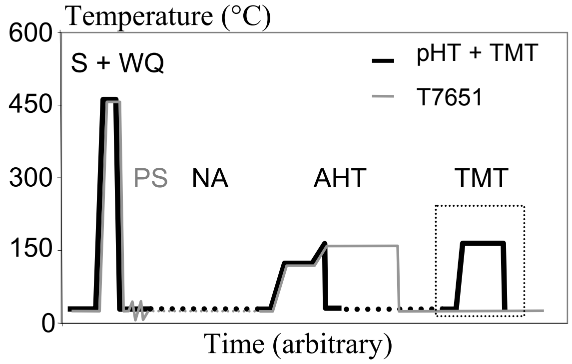

Figure 4. Complete thermal history of a T7651 temper (grey) and of the thermomechanical samples (black).

Results

Creep test on temper T7651

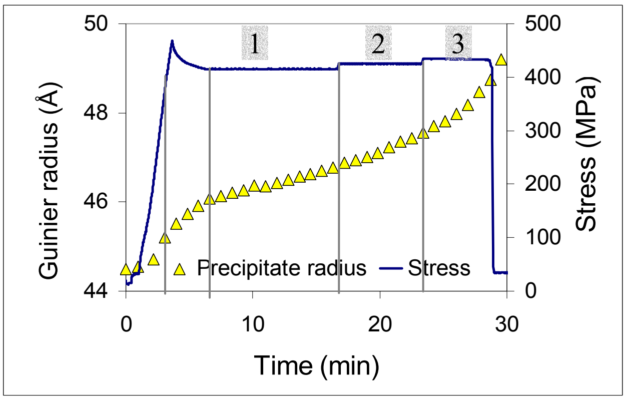

Figure 5. Effect of creep stress jumps on the precipitate radius evolution at 150°C.

Stage	1	2	3
Creep stress (MPa)	415	425	435
Creep rate (s^{-1})	2.05	4.62	14.36

Table 1. Effect of creep stress on creep rate at 150°C.

If the creep rate depends on the creep stress level, the precipitate growth rate is also strongly affected.

Tensile test on a partially aged temper

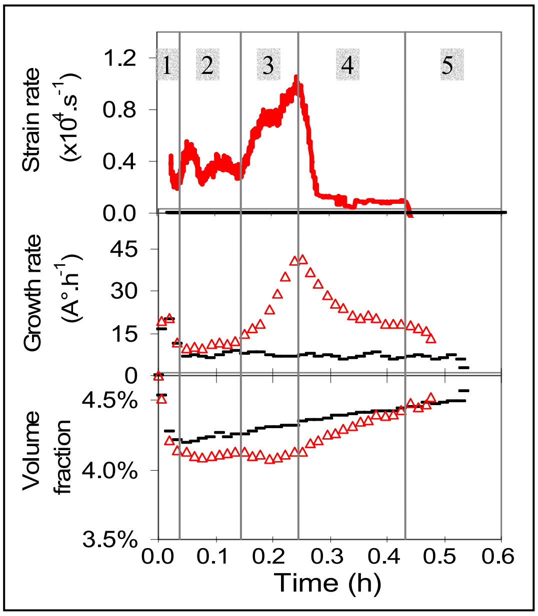

Figure 6. Evolution of the strain rate (imposed parameter) and consequence on the precipitate growth rate and the precipitate volume fraction during a tensile test (red curve), as compared to a stress-free sample (black curve). Initial temper: partial heat treatment (see fig. 4). TMT temperature: 160°C.

The TMT can be separated into 5 stages: heating (1), elastic loading (2), plastic loading (3), relaxation (4) and unloading (5).

Each of those stages affects precipitation in a different way. One of the most striking results is that the precipitate growth rate is maximum at the end of the plastic stage and decreases then, while it remains constant for the stress-free sample. This has been shown on a the various initial tempers and TMT conditions.

Conclusion

We have evidenced that precipitation kinetics is strongly affected by simultaneous forming and ageing: there is a cumulative effect of strain rate and strain: the more strain has been applied, the higher is the dislocation density and the faster will grow the precipitates (static effect), in particular when dislocations are moving (dynamic effect).

Those results have been presented at ICAA11 in Aachen (Germany) in September 2008 [11].

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