Conducting Controlled In Situ High Temperature Tensile Tests

Testing of Mg-alloys within a Scanning Electron Microscope

A methodology to perform in situ high temperature tensile tests of Mg-alloys within a SEM with a high degree of mechanical and temperature control is described in detail. The fact that Mg might vaporize under vacuum requires technical challenges to be overcome if one aims at conducting tests without damaging the microscope. The detailed methodology enabled insights into the link between the development of local strain heterogeneities and the occurrence of fundamental deformation mechanisms.

Introduction

Despite their low density, the use of magnesium alloys in industry is still limited. This is partly due to their poor workability resulting from the hexagonal close-packed structure. Indeed, at room temperature (RT), magnesium and its alloys suffer from their plastic anisotropy deforming essentially by basal slip and twinning, which limits their ductility. RT deformation of Mg alloys has been extensively studied, see e.g. [1-8] among others. It is generally considered that twinning activity decreases and additional slip systems contribute significantly to plastic deformation for temperatures typically higher than 200°C [7, 9]. When the average grain size is small enough (typically about 10 μm) and the strain rate properly controlled, formability can still be increased since superplastic properties can be achieved [10-12]. In the superplastic regime, grain boundary sliding (GBS) is supposed to be the predominant deformation mechanism. However, up to now, the contributions of the different deformation mechanisms as a function of temperature and strain rate are not clearly established.

In order to tackle this question, an AZ31 magnesium alloy was tested under tension in a SEM at various temperatures (up to 300 °C) and strain rates. Digital Image Correlation (DIC) techniques were used to quantify the in-plane strain field. Interpreting these results, however, requires control of the temperature during the in situ tensile test in the SEM, which remains a challenging issue. In the particular
case of Mg alloys, possible evaporation of magnesium in the SEM can also be an issue that has to be overcome. For all these reasons, the study of high temperature deformation of Mg alloys in SEM via DIC techniques remains today poorly documented [13].

At room temperature (RT) few studies can be found in the literature [14-18].

The first objective is to describe in details the experimental procedures to carry out SEM in situ high temperature tensile tests of Mg-alloys with a high degree of mechanical (strain and load) and temperature control without risking to damage the SEM. The second objective is to show through some examples what can be inferred from those experiments to better understand the local mechanical behavior of Mg-alloys and to identify the underlying fundamental deformation mechanisms in the high temperature regime.

Materials and Methods

Material and Sample Preparation
The material investigated was a AZ31 Mg-alloy (3 %wt Al, 1 %wt Zn and 0.4 %wt Mn) sheet with an initial thickness of 2 mm. Micro-tensile specimens with a specific geometry were extracted from the annealed 2 mm-sheet by Electron-Discharge Machining (EDM) with the tensile axis parallel to the rolling direction (RD).

Prior mechanical loading and to enable microstructural investigations, the micro-tensile specimens were prepared by mechanical grinding, followed by diamond polishing (successively 3 and 1 μm) and alumina polishing (0.3 μm). The sample was finally electro-polished at 20 °C in a solution consisting of phosphoric acid (60 %) and ethanol (40 %). The as-received microstructure was fully recrystallized with an average grain size approximately 10-15 μm.

In Situ High Temperature Tensile Testing
The in situ tensile tests were performed with a Gatan Microtest 5000W micro-tensile stage (fig. 1a) within a Zeiss GeminiSEM 500 Field Emission Scanning Electron Microscope. Combining in situ high temperature tensile tests with a high resolution FEG-SEM is also challenging. A specific sub-stage, shown in figure 1b, was developed to set up the micro-machine into the SEM (fig. 1c) while ensuring a good stability during image acquisition and avoiding to damage the rotation motion of the SEM stage.

Zeiss GeminiSEM 500 has unique imaging performance thanks to its new designed electronic column equipped with the Nano-Twin objective lens and a beam booster.
The combination of both enables to get a more efficient collection of secondary electron (SE) when using the In-lens SE detector even if the working distance is large. Here, the working distance was equal to 14 mm when sample was mounted in the micro tensile device. Moreover, the electron optical design of the new lens limits spherical and chromatic aberrations and allows having a very high resolution [19]. The GeminiSEM 500 offers the possibility to work, not only in a high vacuum mode (HV mode, typically $10^{-6}$ mbar), but also in a low pressure mode up to 150 Pa. In this case, the collection of the secondary electrons is done with a specific detector using conversion electrons-photons inside gas (VPSE detector). In summary, the microscope offers high flexibility and versatility for a wide range of applications.

The initial load cell of 5000 N was substituted by a 500 N load cell more suitable for high temperature tensile testing. The commercial software monitoring the microtensile stage enabled only to control the displacement rate. When dealing with high temperature deformation, it is more appropriate to impose a constant strain rate rather than a constant displacement rate. Therefore, a homemade LabView interface was specifically developed allowing to control the strain rate by adjusting the displacement rate throughout the test. Strain rates between $3.10^{-5}$ to $7.10^{-3}$ s$^{-1}$ could be achieved.

In situ tensile tests were carried out between room temperature and 300°C. Controlling the temperature is crucial in the high temperature regime because a temperature change might have a significant impact of the flow stress. In particular, it is important to make sure that the temperature is kept constant ±5°C during mechanical loading. As a result, contact heating systems were prohibited because the contact between the heating element and the specimen is likely to be deteriorated under loading. Rather, we took advantage of the heating jaws so that the contact between the specimen and the heating jaws is maintained throughout the test. A thermocouple located within the jaws enabled the temperature to be controlled using a Proportional Integral–Derivative regulation (PID). An additional thermocouple was spot-welded to the gauge length of the micro-tensile specimen to better reflect the temperature evolution of the region of interest during mechanical testing. The difference between the temperature imposed at the jaws and the temperature measured in the gauge length never exceeded 10°C.

Finally, it turns out important to highlight that two peculiar cautions have to be taken when testing Mg-alloys at high temperature within a SEM.

**Caution 1: The Atmosphere**

When working under HV mode, one has to keep in mind that Mg possesses a vapor pressure of about $10^{-7}$ mbar at 200 °C and $3.10^{-4}$ mbar at 300°C. It means that
given the fact that the HV mode of the SEM is in the range $10^{-5}$-$10^{-6}$ mbar, conducting in situ high temperature tensile test at 300 °C must be prohibited because Mg might vaporize within the chamber and contaminate both the detectors and the column. As a result, HV mode was used between room temperature up to 250 °C. When higher temperatures ($T > 250$ °C) were required, the variable pressure mode (VP mode) was selected in the range of 10 to 30 Pa so as to avoid Mg-vaporization. Moreover, the atmosphere used was nitrogen, in order to avoid the Mg-oxidation. It has to be highlighted that the use of the VP mode changes the image contrast as shown in figure 2. Nevertheless, the DIC routine still works with VPSE images.

**Caution 2: Heating of the Bottom of the Objective Lens**

When working under VP mode, the bottom of the objective lens can be heated to temperatures higher than 80-100 °C because heat conduction is improved when vacuum is deteriorated. Such temperatures over hours can damage the lenses. Consequently, a heat shield made of copper was specifically designed so as to protect the objective lens.

**Micromechanical Characterization**

Prior to mechanical loading, a region of interest located within the gauge length of the micro-tensile specimens was selected and characterized by EBSD (EDAX system) using a step size of 0.5 μm. Microtexture as well as grain boundary positions can be determined from those data.

Microscale strain measurements by DIC require SEM images to exhibit a sufficient local contrast at the grain scale. However, because of the too low contrast shown by the AZ31 alloy: only the second-phase particles can provide contrast but its density does not allow enough measurements locally. Local contrast was generated by depositing fiducial microgrids onto the region of interest that was preliminary characterized by EBSD along the gauge length of the specimen. Microgrids were deposited either by FIB (Pt grids) or by Electron beam lithography (Au grids). For the microgrids fabricated by FIB, a Zeiss NVision40 CrossBeam SEM equipped with a FIBICS external scan generator and Nano Patterning and Visualization Engine (NPVE) software was used. The Pt organo-metallic was cracked under FIB with a current of 10 pA under 30 kV. The global dose per line was around 3.3 nC/μm$^2$. For the ones deposited by E-beam lithography, they were made using the procedures detailed in [16, 17]. Gold and Platinum were chosen because of their ductility, good contrast relative to magnesium in secondary electron imaging, and thermal stability in the range of temperature investigated in the present work. The typical grid size was 2 or 1 μm with line width of 200-300 nm produced over area of 100 x 100 μm.
In the undeformed configuration and after each strain increment, a high definition image (4096 X 3072 pixels) was acquired using SE between RT to 250°C and Variable Pressure SE for T > 250°C. To enable DIC to be performed, the images must not show too much noise and should exhibit a good local contrast. In order to reduce the noise in the images, slow scanning speeds were chosen leading a time of about 10-15 minutes to acquire an image. The grayscale histogram was optimized by adjusting the brightness and contrast so as to cover the whole grayscale, i.e. 0-255.

Comparison between the undeformed and deformed microgrids enable to determine quantitatively the in-plane displacement field. To do that, DIC was employed using the software called CMV. The reader is referred to the References [20-23] for the DIC procedures. DIC was run with a 30 x 30 pixels correlation subset. Within the correlation subset, a bilinear interpolation of grayscale was used to allow subpixel accuracy of displacement to be achieved.

To establish correlations between local strain and microstructure/microtexture, the strain maps (obtained by DIC from the SEM images) and the associated orientation imaging map (obtained by EBSD) must be superimposed. Because of distortions and magnification/rotation variations, the superimposition of the raw images gave a poor correlation. To correct this, the transformation between both images was computed. From a given set of reference points (about 20 points) easily distinguishable in the EBSD map as well as in the SEM (undeformed microgrids) image, the transformation matrix was fully determined. This transformation takes into account magnification, distortion and rotation variations. Once determined, the transformation was applied and a good matching was achieved.

**Examples of Strain Heterogeneities Initial Microstructure and Microtexture**

The initial texture exhibits a strong basal texture (fig. 3b) typically observed for the AZ31 alloy [10, 13]. In other words, it means that the hexagonal lattice has its c-axis preferentially oriented long the ND direction of the metal sheet whereas no preferential orientation was observed in the (RD, TD) plane. The average grain size was found to be about 12 μm.

**Mechanical Behavior**

Determining the true stress-true strain behavior during *in situ* tensile tests at high temperature is not straightforward. Indeed, the displacements and therefore the strain cannot be estimated accurately because it would require the use of a local extensometer attached to the gauge length of the sample during high temperature deformation. Rather the tensile true stress-true strain behavior was extracted from
the force measured by the load cell, and the average tensile strain computed over the gridded region based on the local tensile strain $\varepsilon_{11}$ measured at each grid intersection. At each increment of displacement corresponding to an image acquisition, stress relaxation occurred. The true stress was extracted just before the stress release. The associated true strain was directly extracted from the DIC measurements. The true stress-true strain tensile behavior is displayed in figure 4.

**Quantification of the Strain Heterogeneities and Underlying Deformation Mechanisms**

The evolution of the deformation of the microgrids during an *in situ* tensile test performed at $3.10^{-5}$ s$^{-1}$ at 250 °C is illustrated in figure 5a-b. Those high-definition images were acquired using the secondary electron (SE) contrast after different macroscopic tensile strains. The corresponding tensile strain maps calculated by DIC are shown in figure 5c-d where the location of the grain boundaries (misorientation > 15°) are also highlighted in white. The latter were identified based on the EBSD measurements performed on the undeformed configuration. The strain maps are displayed as color maps corresponding to the magnitude of the local tensile strain.

The development of spatial heterogeneities of the plastic tensile strain $\varepsilon_{11}$ within the region containing the microgrids is clearly revealed. Some regions exhibit local tensile strains 3 to 4 times larger than the macroscopic tensile strain while other regions are significantly less distorted in comparison with the macroscopic tensile strain.

The methodology suggested in the present contribution enables to better understand how local strain heterogeneities are distributed within the microstructure and to identify the underlying deformation mechanisms. An example is given in figure 6 after 5 % of macroscopic strain. Several regions where the local strain is higher than the average are pointed out to identify what are the underlying deformation mechanisms responsible of those strain heterogeneities. As illustrated by figure 6a, the intragranular strain within some grains was very low, the microgrids being just rotated while strain amplification was sometimes found due to the occurrence of grain boundary sliding, see figure 6b. In some regions, the microgrids were strongly distorted, the initial square grid being sheared, most likely because of the occurrence of dislocation slip as illustrated in figure 6c.

**Summary and Conclusions**

*In situ* high temperature tensile tests within a FEG-SEM is a powerful characterization method since one can benefit from both microstructural and mechanical inputs to shed light on the mechanical behavior of Mg-alloys relying on
fundamental mechanism of deformation. A rigorous methodology to run those experiments was described in detail. Those in situ tensile tests should provide new quantitative insights into the development of strain heterogeneities at the microscale. They have quantitatively confirmed that grain boundary sliding can be significantly activated when the temperature reaches 250°C.

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More information on SEM

References


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