Evidence of damage evolution during creep of Al–Mg alloy using synchrotron X-ray refraction

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In order to provide further evidence of damage mechanisms predicted by the recent solid-state transformation creep (SSTC) model, direct observation of damage accumulation during creep of Al–3.85Mg was made using synchrotron X-ray refraction. X-ray refraction techniques detect the internal specific surface (*i.e.* surface per unit volume) on a length scale comparable to the specimen size, but with microscopic sensitivity. A significant rise in the internal specific surface with increasing creep time was observed, providing evidence for the creation of a fine grain substructure, as predicted by the SSTC model. This substructure was also observed by scanning electron microscopy.

1. Introduction

Although cavities and pores are present in metallic materials from their manufacture (Toda *et al.*, 2013; Kassner & Hayes, 2003), it is well established that creep introduces further significant damage. In fact, the nucleation and growth of cavities typically appears even from the first stages of creep (Toda *et al.*, 2013). The size of the cavities ranges from 0.1 to 10 μm (Balluffi & Seigle, 1957; Bouchard *et al.*, 2004). Creep damage distribution is greatly influenced by microstructural features such as grain boundaries, precipitates or second phase particles (Toda *et al.*, 2013). Several mechanisms have been proposed for cavity nucleation at grain boundaries. Among them, vacancy accumulation, grain-boundary sliding and the existence of dislocation pile-ups are the most widely accepted (Toda *et al.*, 2013). Similarly, several mechanisms that control cavity growth have also been suggested: grain-boundary diffusion, grain-boundary sliding, constrained diffusion and plastic deformation (Toda *et al.*, 2013). In addition, under particular conditions, simultaneous diffusion and plastic deformation have been considered as a source of cavity growth during creep (Toda *et al.*, 2013). During the final stages of creep, cavity coalescence forms cracks, which then propagate to cause failure. The size and distribution of cavities depend on the stress state (Camin, 2014). Tomographic and diffraction studies on brass have shown that their volume grows exponentially with creep time (Pyzalla *et al.*, 2005). At high stresses in commercial alloys, cavities can nucleate both at grain boundaries and within the grains (Fuller *et al.*, 1999), preferentially at particles or inclusions. In the case of high-purity metals such as 99.999% Al, cavities do not generally form (Toda *et al.*, 2013). However, cavitation is observed in lower-purity metals such as 99% Al (Yavari & Langdon, 1983). In the case of class A alloys (Sherby & Burke, 1968), such as

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the Al–Mg alloy discussed here, it has been suggested that a dislocation forest structure is developed during creep at medium and low stresses. This special structure should influence the spatial and size distribution of cavities. However, no direct observation of damage accumulation has been provided in the Al–Mg system (Yavari *et al.*, 1981).

In this study, we show for the first time creep damage accumulation in an Al–Mg alloy by means of X-ray refraction techniques. These techniques (Hentschel *et al.*, 1987) have been successfully used to detect inhomogeneities (pores, cracks, particles *etc.*) in the bulk of light materials such as ceramics (Kupsch *et al.*, 2017; Müller *et al.*, 2018) and epoxy composites (Harbich *et al.*, 2001). Recently, such techniques have been extended (with the use of synchrotron radiation) to metal matrix composites (Müller & Hentschel, 2004) and even to light alloys (Laquai *et al.*, 2015, 2018).

Scanning electron microscopy (SEM) was also used to corroborate the X-ray refraction data. We finally demonstrate that the present observations are in agreement with the damage accumulation that is predicted in the recent solid-state transformation creep (SSTC) model based on isothermal transformation processes (Fernández *et al.*, 2016). Note that the SSTC model describes creep of aluminium alloys at the same length scale as X-ray refraction, instead of using the traditional approach based on micromechanisms. In this SSTC model, the existence of cavities is a natural consequence of dislocation motion and accumulation at grain and subgrain boundaries.

2. X-ray refraction

X-ray refraction techniques (Hentschel *et al.*, 1987) were introduced a couple of decades ago, and have been successfully used for both materials characterization and non-destructive testing (Müller & Hentschel, 2013). X-ray refraction techniques yield a relative internal specific surface (*i.e.* surface per unit volume, relative to a reference or initial state). The smallest detectable object size is as low as 1 nm, and therefore a population of objects is necessary to yield an integrated signal above the background noise. These techniques are extremely well suited to investigating defects such as cracks and pores. Furthermore, X-ray refraction techniques are sensitive to defect orientation, thereby allowing the identification of different kinds of defects. The refraction signal of an isotropic inhomogeneity (*e.g.* spherical pores) will be present in any orientation of the specimen with respect to the detection system, whereas for cracks or elongated pores the signal vanishes when the defect surface normal is not parallel to the scattering vector.

X-ray refraction occurs when X-rays interact with interfaces between materials of different densities, as in the case of cracks, pores and particles in a matrix. This is analogous to the behavior of visible light in transparent materials, such as lenses or prisms. The difference in refraction indices of the two interfacing materials (the so-called refraction decrement) determines both the refraction angle at the interface and the refracted intensity, and is dependent on the wavelength of the

Table 1
Chemical composition of the studied Al–Mg alloy.

Mg	Fe	Cu	Mn	Si	Cr	Ni	Zn	Ti
3.84 ± 0.02	0.010	0.005	0.003	0.06	0.001	0.003	0.003	≤0.010

radiation. Consequently, X-ray optical effects can be observed at small scattering angles (between several arcseconds and a few arcminutes), as the refraction decrement for X-ray radiation is of the order of 10^{-5} . Since the typical X-ray wavelengths lie around 0.1 nm, X-ray refraction is sensitive to inner surfaces and interfaces of defects (*e.g.* pores and cracks) with nanometric dimensions. The detectability is not to be confused with the spatial resolution of the techniques. The spatial resolution is limited by the beam size for laboratory setups and by the pixel size of the detector system at a synchrotron beamline. It must be emphasized that, because of the inevitable background noise, it is impossible to detect (and *a fortiori* image) one single defect. As mentioned above, the defect population has to be large enough to produce a refraction signal above the background noise. X-ray refraction is mostly used in radiographic mode and the specimen is typically a platelet. This yields a 2.5-dimensional picture, *i.e.* two-dimensional but integrated over the specimen thickness. Therefore, a population of defects is detected, rather than single defects being imaged. Unlike some imaging techniques such as radiography and perhaps grating interferometer imaging (Pfeiffer *et al.*, 2009), the X-ray refraction signal can be quantitatively correlated to microstructural changes and micromechanical models (Cooper *et al.*, 2017). Synchrotron X-ray refraction radiography is a method utilizing the concept of analyzer-based imaging (ABI), also known as diffraction-enhanced imaging (Chapman *et al.*, 1997). In ABI, information about the microstructure of the specimen is obtained from images taken at different positions of an analyzer crystal on the so-called rocking curve. A detailed description of the data analysis of ABI is given in previous work (Oltulu *et al.*, 2003; Zhang *et al.*, 2008).

3. Experimental

3.1. Materials and mechanical tests

The chemical composition of the studied alloy is summarized in Table 1. The microstructure of the materials is shown in Fig. 1. The average grain size is between 60 and 70 µm. Cylindrical specimens of the Al–Mg alloy were subjected to tensile creep tests at temperature $T = 573$ K and stress $\sigma = 13.8$ MPa. A heating rate of 100 K h^{-1} was applied until the creep testing temperature was achieved, following previous work (Fernández & González-Doncel, 2007). Constant tensile stress during specimen elongation was guaranteed by means of a so-called Andrade cam (Fernández *et al.*, 2016), which reduces the applied load according to the specimen section reduction. Sister specimens were exposed to creep conditions for $t = 0$ s, $t = 88\,000$ s and $t = 194\,000$ s, corresponding to

nominal strains of 0, 0.13 and 0.29, respectively. These specimens will be named e0, e13 and e29 in the remainder of the paper.

3.2. X-ray refraction radiography

The creep-tested specimens were ground on both sides, and successively polished with 9, 3 and 1 μm diamond paste, in order to obtain platelets about 1 mm thick.

X-ray refraction radiography measurements were carried out at the BAM synchrotron station BAMline at Helmholtz-Zentrum Berlin, Germany (Müller *et al.*, 2009; Görner *et al.*, 2001). The three specimens were mounted side by side in a slide frame (Fig. 2). A double-crystal Si(111) monochromator (DCM) was used to extract a highly collimated monochromatic X-ray beam (energy band width 0.2%). The beam energy was set to 17.5 keV to achieve a specimen X-ray transmission of about 30%. A Princeton Instruments camera

Figure 1
SEM image of a transverse section of the as-fabricated specimen.

Figure 2
X-ray refraction experimental setup at the BAMline. The blue arrows indicate the beam path. The specimens are mounted in a slide frame (right). A sketch of the setup is also included below the photograph.

(1340 $\times$ 1300 pixels) in combination with a lens system and a 50 μm thick CdWO_4 scintillator screen provided a pixel size of $5.3 \times 5.3 \mu\text{m}$, capturing a field of view of about $7 \times 7 \text{ mm}$ (Rack *et al.*, 2008). The incident beam was narrowed to the field of view by a slit system in order to avoid detector backlighting (Lange *et al.*, 2012). In contrast to transmission-based radiographic measurements, an Si(111) analyzer crystal was placed in the beam path between the specimen and the camera system (see sketch and photograph in Fig. 2). The analyzer crystal reflects the beam coming out of the specimen into the detector system if the Bragg condition for the Si(111) plane is fulfilled. By tilting the analyzer crystal around the Bragg angle of the analyzer crystal ($\theta_B = 6.487^\circ$ at 17.5 keV) the so-called rocking curve is recorded. This describes the reflected beam intensity as a function of the deviation from the Bragg angle ($\Delta\theta = \theta - \theta_B$). The rocking curve was recorded for each specimen by taking 41 radiographs between $\theta = 6.483^\circ$ and $\theta = 6.491^\circ$ (with a step width of $\delta\theta = 0.0002^\circ$). The $7 \times 7 \text{ mm}$ field of view allowed exposure of two specimens simultaneously. Thus, the three specimens could be imaged in two successive measurements, whereby specimen e13 was imaged twice and could be used to calibrate the two measurements. All specimens were measured in vertical and horizontal orientation (*i.e.* with the scattering vector parallel and perpendicular to the creep load axis, respectively). To correct for the detector dark current and readout noise, an image without the X-ray beam was acquired.

3.3. Data evaluation

The 41 radiographs taken between $\theta = 6.483^\circ$ and $\theta = 6.491^\circ$ with and without the specimen in the beam were analyzed by in-house software based on LabView (National Instruments Corporation, Austin, Texas, USA). Fig. 3 shows the rocking curves extracted from one arbitrary detector pixel with (open circles) and without the specimen in the beam (filled circles).

Figure 3
Rocking curves measured at one arbitrary detector pixel. Filled circles: without specimen; open circles: with specimen. The green shaded area represents the rocking curve (calculated, not measured), assuming the specimen does not have any internal interfaces.

The rocking curve without the specimen represents the intrinsic beam divergence (FWHM = $0.9''$, *i.e.* 0.2% energy bandwidth) and the overall beam intensity (integral of the curve). The rocking curve with specimen has an increased width (FWHM = $3.1''$) as a result of refraction at interfaces inside the specimen. In contrast to the aforementioned work (Oltulu *et al.*, 2003; Zhang *et al.*, 2008) for the practical use of damage quantification, we use the expression ‘X-ray refraction’ for all deviated X-rays (*i.e.* ultra-small-angle scattering and refraction signal caused by the inner structure of the specimen) in the rest of this paper. Without interfaces, and therefore without refraction, the light green curve sketched in Fig. 3 would have been measured. The analysis software delivers the following rocking curve values: peak integral, peak height, peak position and FWHM for each pixel in the image. The peak height corresponds to the refraction prop-

erties of the specimen, and the peak integral relates to the absorption properties.

The values of the peak height and peak integral are then represented as gray values in separate two-dimensional images for further evaluation (see Fig. 4).

The data treatment (performed with the software *Fiji Image J*; Schindelin *et al.*, 2012) consisted of calculating the attenuation images [$\mu d = -\ln(I/I_0)$] (Fig. 5) and the quantity $C_m d$ (Fig. 5*b*), where d is the specimen thickness and C_m is the so-called refraction value. The refraction value was calculated using (1) [for the derivation of the formula see the appendix in the article by Nellesen *et al.* (2018)]:

$$C_m d = 1 - \frac{I_R}{I_{R0}} \frac{I_0}{I}, \quad (1)$$

where I_R and I_{R0} are the rocking curve peak intensity with and without the specimen in the beam and I and I_0 are the integrated intensity of the rocking curve with and without the specimen in the beam, respectively. The influence of the specimen thickness d is eliminated by dividing the refraction by the transmission images. This yields the relative specific refraction value C_m/μ in Fig. 5(*c*), which correlates to the relative specific internal surface of the specimen.

4. Results

Fig. 6 shows transmission images used to analyze pores and particles. Dark spots are associated with pores and cracks, while bright spots should indicate objects denser than the matrix. However, the actual situation is more complex, as will be discussed below. Fig. 6 shows the increase of porosity with creep time, particularly for the e29 specimens [see features on the left side of specimen e29 in Fig. 6(*c*)]. Furthermore, with increasing creep time, the bright features evolve from diffuse spots to sharp dots. The presence of sporadic cracks for specimens e0 (Fig. 6*a*) and e13 (Fig. 6*b*) must be related to the fabrication conditions (elongated extrusion defects in the middle of the bar). The average value of the linear absorption coefficient does not vary as a function of creep time (Fig. 7).

The specific refraction value (C_m/μ) maps are shown in Fig. 8 for the two specimen orientations. Although some cracks are observed in the e0 and e13 specimens, we qualitatively observe an increase in C_m/μ as a function of time. This indicates an increased number of internal defects (Harbich *et al.*, 2001; Hampe *et al.*, 1999; Hentschel *et al.*, 1998; Kupsch *et al.*, 2017).

The results of the quantitative analyses of the images in Fig. 8 are presented in Fig. 9. Here, the inhomogeneity of the specific surface value is also shown.

Fig. 9 conveys a few important messages:

(i) The amount of initial internal specific surface (thereby damage,

Figure 4
Two-dimensional matrix given as grayscale images for rocking curve peak height (top) and peak integral (bottom) with (left) and without (right) the specimen.

Figure 5
Example of the data treatment process (specimen e13 left; specimen e29 right): (a) attenuation image (μd), (b) refraction value ($C_m d$) and (c) relative specific refraction value (C_m/μ), proportional to the internal specific surface.

analogous to the work of Harbich *et al.*, 2001; Hampe *et al.*, 1999; Hentschel *et al.*, 1998; Kupsch *et al.*, 2017) increases linearly with creep time.

(ii) Interestingly, C_m/μ is not zero in the uncrept conditions. This confirms the presence of defects in the as-fabricated material (see §1).

(iii) The internal specific surface is consistently larger in the horizontal specimen mount than in the vertical mount. This indicates that the detected defects are oriented along the extrusion or creep direction.

(iv) Defects are not homogeneously distributed within the specimens [see Figs. 9(b) and 8, where defects are observed to be predominantly aligned in bands along the extrusion axis]. To quantify how inhomogeneous damage distributes over each specimen, the width of the distribution of the internal specific surface was calculated as the standard deviation of the

refraction value over the whole of each specimen. This defines the inhomogeneity plotted in Fig. 9(b). The inhomogeneity carries the same units as the specific surface.

(v) The slope of the C_m/μ curves *versus* creep time for the horizontal specimen mount is larger than that for the vertical one. This indicates that the degree of defect alignment along

Figure 6
 μd images (vertical specimen orientation). (a) e0, (b) e13, (c) e29. Pores and macro cracks are indicated by arrows.

Figure 7
Average linear absorption coefficient of the investigated specimens at 17.5 keV photon energy.

Figure 8
Images in a vertical specimen mount (a) e0, (b) e13, (c) e29, and horizontal specimen mount (d) e0, (e) e13, (f) e29.

Figure 9
Average specific surface value C_m/μ (a) and its inhomogeneity across the specimen (b) for all specimens in a horizontal and a vertical mount. In (a) lines are linear fits and in (b) they are guides for the eye.

the creep load axis increases with creep time. This is related to the extrusion process and material texture.

5. Discussion

In Fig. 6 we observed bright spots of apparent high density. Fig. 10 compares the attenuation coefficient of specimen e29 in horizontal and vertical mounts. Many of the features that can be seen in one orientation cannot be seen in the other (highlighted by the yellow circles). This effect can be explained in terms of diffraction by Al grains; when a grain fulfills the Bragg condition, it removes intensity from the transmitted beam and therefore appears as absorbing.

Hence, the bright spots in Fig. 10 correspond to Al grains in a specific orientation with respect to the monochromatic X-ray beam. We have mentioned that these spots become sharper and smaller and additional small spots appear with creep time. This calls for the formation of substructures of much smaller size than in the case of Al6061 and pure Al reported by Fernández *et al.* (2016) (for an equivalent stress). In other words, the main difference between the microstructural features in the alloys studied by Fernández *et al.* (2016) and the alloy studied here is the length scale of the substructure; in Al6061 the subgrain size is about 20–50 μm (Fernández *et al.*, 2016), whereas the structures formed here are possibly about 200 nm in size. Magnesium introduces a strong distortion in the Al lattice and this explains the fact that in specimen e0 the spots appear diffuse. As creep progresses, the dislocation network present under the initial conditions rearranges to form substructures, and therefore the diffuse spots become sharper – *i.e.* have higher contrast – and more small spots appear. This scenario is equivalent to the statement made above, that there is continuous formation of damage features (cavities) of a size equivalent to the substructure, corresponding to the increasing refraction signal as a function of

creep strain (see Fig. 9a). This is consistent with the fact that creep curves of the present Al–Mg alloy appear very different from those shown by Fernández *et al.* (2016) (see the sketch in Fig. 11). At the same deformation ε_1 (horizontal dashed line), damage is the same in the two alloys, but distributed according to the substructure length scale. Consequently, ε_1 is reached at a very different creep time under the same experimental conditions (of applied stress and temperature).

This implies that, in the tertiary creep stage, ductility is more pronounced in Al–Mg than Al6061, and Al–Mg would break at much larger strain. If the substructures have a smaller length scale, the propagation of macroscopic cracks would be much more difficult than in the case of a coarser microstructure. In fact, the fracture toughness is inversely proportional to grain size.

The fact that damage forms at a finer scale in the Al–Mg alloy (than in pure Al) is visible in the SEM images in Fig. 12. In the initial conditions, large cavities are already present at grain boundaries (Fig. 12a). Those cavities represent dislocation accumulation caused by the extrusion process. During creep, dislocation motion and rearrangement occurs, also involving those dislocations initially located at cavities. Consequently, this rearrangement process suppresses large cavities located at grain boundaries, and favors formation of small cavities inside the grains (Figs. 12b and 12c). The large cavities present in the initial conditions explain the nonzero refraction value (Figs. 8a, 8d and 9a). The appearance of small features to the detriment of large cavities explains the increase in the refraction value; the internal specific surface is inversely proportional to the dimensions of the features (Harbich *et al.*, 2001; Hentschel *et al.*, 1998; Kupsch *et al.*, 2017).

The present observations from X-ray refraction, showing a very fine structure of cavities, are in good agreement with the creep damage accumulation expected from the SSTC model (Fernández *et al.*, 2016). One of the most relevant characteristics of this model is that creep of aluminium alloys is described at the mesoscale. Alloys that are traditionally considered to form subgrains during creep (Al 99.8% and Al6061) and those traditionally considered to form dislocation forests (Al–Mg) are harmonized in the frame of the SSTC model. Therefore, cavities for the present Al–Mg alloy are

Figure 10
Comparison of images for specimen e29 in (a) horizontal and (b) vertical mounts.

Figure 11
Sketch of typical creep strain *versus* time experimental curves for Al6061 (dashed line) and the Al–Mg alloy (solid line), under the same conditions of applied stress and temperature. Secondary creep is very short in Al–Mg. Straight lines indicate the minimum creep strain rate. The horizontal line indicates a given strain, which is attained at different times (t_1 and t_2) depending on the alloy. This implies different damage distribution in the alloys.

expected to be formed by dislocation motion and accumulation at grain, and mostly subgrain, boundaries. This view describes the traditional concept of the microstructure (*i.e.* as dislocation forests in this alloy) as a very fine subgrain structure. The fine damage structure observed by X-ray refraction and SEM, instead of the delocalized damage represented by dislocation forests, validates the hypothesis that the structures formed during creep of Al–Mg alloys correspond to small ($<1\ \mu\text{m}$) subgrains.

In line with current investigations (Fernández *et al.*, 2016), future work will be focused on the comparison of damage patterns developed in alloys traditionally considered as forming subgrains during creep (Al 99.8% and Al6061) and the alloy studied in this work (Al–Mg), which is classically considered not to form subgrains (but instead dislocation forests). This comparison would provide new insight into the

mesoscale description of the creep phenomenon in aluminium alloys.

6. Conclusions

Synchrotron X-ray refraction proved to be a useful technique to quantitatively determine the evolution of damage during creep. In particular, for the case of Al–Mg class A alloys, it allowed us to quantify damage at a finer scale than the grain size, yet over macroscopic (specimen) dimensions. In fact, we observed that the number of fine ($<1\ \mu\text{m}$) defects in Al–Mg increases with creep strain. These defects are traditionally described as dislocation forests, but X-ray refraction data showed that they can be considered as small substructures, as implied by the solid-state transformation creep (SSTC) model. The evolution of these substructures was quantified to be linear with creep time. By means of X-ray refraction radiography, we also observed that damage is concentrated in bands aligned with the creep load (and with the extrusion) axis. These findings were confirmed by SEM images, where the appearance of small-scale defects within grains and the disappearance of large cavities at grain boundaries was observed.

Since the present findings confirm and complement our current model that associates damage with the formation of substructures, we aim to extend this study to include alloys that can develop subgrains (such as fine grain Al6061 or coarse grain Al).

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Figure 12
SEM images of specimens e0 (a), e13 (b), e29 (c) showing cavities associated with the substructure formed during creep of Al–Mg: (a) intergranular voids in the initial state; (b), (c) formation and growth of intragranular cavities.

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