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On the damage and fracture of commercially pure magnesium using x-ray microtomography

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ABSTRACT

The nucleation, growth and linkage of microvoids are examined in commercially pure magnesium using x-ray computed microtomography (XCT). Tensile testing was performed inside the chamber of an XCT system with each test stopped at various levels of deformation to acquire scans. The nucleation of flat penny-shaped voids is observed relatively early during plastic deformation due to failure at twin and grain boundaries. Once nucleated the voids exhibit rapid growth along preferential directions related to the crystallographic texture of the material as well as the sample geometry. The final fracture occurs by a macroscopic shearing process. Furthermore, the fracture surface reveals two main characteristics: a faceted surface associated with twinning and irregular features related to the failure of grain boundaries. The data exhibit both a quantitative and qualitative difference in the damage development process in magnesium as compared with FCC metals such as aluminum and copper.

1. Introduction

Magnesium and its alloys are of significant research interest for their potential use as lightweight structural materials, particularly in the automotive and aerospace industries. Despite its low density and high strength to weight ratio the application of magnesium is currently limited by its relatively poor ductility at room temperature. This in turn can be attributed to the hexagonal closed packed (hcp) crystal structure of magnesium which offers limited slip systems and considerable mechanical anisotropy. It is well established that the crystallographic texture has a significant impact on the deformation of magnesium and its alloys [1–4]. Moreover, there has been extensive work aimed at developing new alloys with improved mechanical behavior [5].

One of the most significant challenges preventing greater utilization of magnesium alloys is the poor understanding of fracture behavior. Ductile fracture in metallic materials occurs by the nucleation, growth and linkage of microvoids within the bulk of the material [6–11]. In our recent work we have largely focused on isolating specific aspects of this process, particularly void growth and coalescence by studying materials (primarily Al, Cu and Mg) with prenucleated voids. This has included the testing of both 2D model materials, which consist of thin sheet tensile samples with holes drilled in the gage section [12–14], and 3D model materials in which voids are embedded within the bulk of the material [15–17]. The holes in the 3D model materials exhibit more realistic constraints and x-ray tomography has been used to observe void growth and linkage in situ. Our study of the growth and coalescence of magnesium shows that the behavior is very different from that observed in fcc metals (Cu and Al) studied previously. Whereas the fcc metals exhibit behavior that is consistent with continuum mechanics models for void growth and coalescence this is not true in magnesium. Instead the behavior is dominated by microstructural effects related to the presence of grain boundaries and the development of twins [13,14,18]. Moreover, this work shows that 2D techniques must be complemented with 3D techniques in order to completely characterize damage and fracture [19].

X-ray tomography provides a non-destructive method for investigating internal processes as they occur and has recently been applied with great success to the study of fracture [20–25]. For example, the early study by Babout et al. [20] used x-ray tomography to investigate the effect of the matrix properties on void nucleation in an aluminum matrix reinforced with hard ceramic particles. Commercially pure aluminum and the 2124 aluminum alloy were used to determine the impact of matrix strength. It was established that particle-matrix decohesion was the dominant mechanism of void nucleation in the soft matrix while particle cracking occurred more frequently in the hard matrix. Lhuissier et al. [26] used x-ray tomography to study the evolution of damage in a wrought AZ31 magnesium alloy during high temperature deformation. This study focused on the nucleation of cavities as well as their interaction with particles. They established that a significant number of voids displayed no apparent relationship

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with intermetallic particles. It should be noted that high temperature deformation induces grain boundary sliding and this is likely the void nucleation mechanism in cases where the voids did not exhibit contact with particles. Nevertheless, the results provide support to experiments by Calhoun and Stoloff [27] who used microscopy to show that void nucleation in magnesium alloys is not always associated with particles.

Lecarme et al. [28] investigated the heterogeneous nature of void growth in a Ti-6Al-4V alloy subjected to uniaxial tension using x-ray microtomography. They developed an algorithm to track the position of cavities at successive increments of deformation, which relies on a graph-based data association approach. The equivalent radii of the voids within the gage region were analyzed and compared with the Rice and Tracey model [8] as modified by Huang [29]. They concluded that the large range of variability observed in the void growth behavior was related to the effects of the local crystallographic orientation. Lhuissier et al. [30], as part of their study on high temperature deformation in a wrought AZ31 magnesium alloy, used x-ray tomography to determine the volume of the 60 largest cavities as a function of the macroscopic strain. They compared the void growth results to a relatively simple expression proposed by Pilling and Ridley [31]. In this case the voids were represented by volumes rather than equivalent radii. An advantage to this expression is that it can be applied to voids with complex shapes. Similar to the results of Lecarme et al. [28], the variability in the growth behavior was quite large. In some cases, void growth was observed with no apparent interaction between neighboring cavities. Their results provide supporting evidence that the local crystallographic orientation has a significant impact on void growth behavior in materials with low crystal symmetry.

Steglich and Morgeneyer [32] used x-ray tomography to study the evolution of damage at room temperature in a commercial AZ31 magnesium alloy. Kahn tear tests were used to observe the crack extension behavior. The radiographs reveal that cracks extend by void sheeting. The results of Barnett et al. [33] and Kang et al. [34] provide supporting evidence to the shear nature of fracture. In these investigations, flat penny shaped voids were observed in the vicinity of the fracture surface. However, the mechanism of void nucleation is not clear.

In this paper we present the results of a study on damage development in commercially pure magnesium using x-ray tomography, coupled with microscopy surface observations, to characterize all aspects of the fracture behavior. The use of pure magnesium, which eliminates void nucleation at particles, enables a clearer test to be made of the conjectures of Calhoun and Stoloff [27] and Lhuissier et al. [26] that voids in magnesium alloys do not need to be nucleated at particles. It also enables us to develop a clearer picture of the role that microstructural inhomogeneity plays in the development of ductile damage in this material.

2. Experimental methods

The material used in this work is hot rolled commercially pure magnesium with an initial thickness of 1.5 mm. Electro discharge machining was used to cut tensile samples with a double dog bone configuration, as illustrated in Fig. 1. The central gage section has a length of 1 mm and a width also of 1 mm. The small gage length is necessitated by the use of tomography which has a limited range of observation. The grinding and polishing procedure used for sample preparation is shown in Table 1.

Following mechanical polishing, samples were annealed at 350 °C for 1 h in a tube furnace purged with argon. A final chemical polishing was then used to remove any oxidation and fine scratches remaining on the specimen surface. The samples were immersed in a 10% solution of nitric acid in anhydrous ethanol for approximately 30 s and then rinsed with ethanol. Material preparation led to a slight variation in the final thickness of the samples; however, on average the thickness was 1 mm. The average grain size measured using the intercept method (ASTM E112 [35]), was approximately 40 μm. Tensile tests were carried out using a Skyscan Material Testing Stage inside the chamber of a Skyscan 1172 microtomography system. The testing speed was held constant at 2 μm/s and the tests were stopped at various levels of deformation to acquire scans for analysis. Radiographs of the gage section were obtained in rotation increments of 0.4° using a voltage of 90 kV and a pixel edge length of 2.77 μm. The radiographs were reconstructed using NRecon software which applies the Feldkamp algorithm for cone beam volumetric reconstructions. Separation of the matrix and voids is achieved in CTAn using a thresholding procedure described by Nemcko [36]. Fig. 1 shows the sample geometry as well as a 3D reconstruction of the gage region after some damage accumulation. The voids are shown in red and the matrix is transparent gray.

Several references will be made to the thin sheet samples described by Nemcko et al. [18] to compare the damage nucleation mechanisms and the fracture surface characteristics. These samples were tested in tension using a MTII/Fullam micro-tensile stage under a Nikon AZ100M stereoscope. A constant crosshead speed of 5 μm/s was used and the test was stopped in increments of deformation to acquire optical micrographs. All of the fracture surfaces in this work were investigated using a JEOL 6610LV SEM.

3. Results

3.1. Void nucleation

Fig. 2 shows optical micrographs of the gage region of thin sheet materials pulled in tension to various levels of deformation. The red and green arrows are used to track the history of selected grain and twin boundaries respectively. At a strain of 0.020, a relatively large step size in the z direction (out of plane) is required to obtain a focused image in the region adjacent to the grain

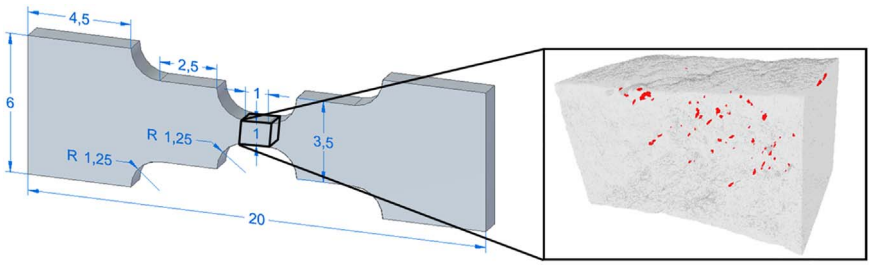

Fig. 1. Tensile sample geometry (dimensions in mm) and 3D reconstruction of the gage section after some deformation and damage. (For interpretation of the references to color in this figure, the reader is referred to the web version of this article.)