

## Cavitation during high-temperature deformation in Al–Mg alloys

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### Abstract

It has recently been revealed that high-density pre-existing hydrogen micropores, formed during production processes, exhibit premature growth and coalescence under external loading at room temperature, thereby inducing ductile fracture. This process is incidentally supplemented by the well-established ductile fracture mechanism based on particle damage. It is reasonable to assume that the pre-existing hydrogen micropores may also contribute to damage evolution at high temperatures. In the present study, synchrotron X-ray microtomography was applied to the in situ observation of deformation and fracture in Al–Mg alloys at a high temperature. High-density hydrogen micropores were observed in the alloys. Flow localization controlled deformation through the mechanism of solute drag creep. A combined effect of grain boundary sliding and heterogeneous nucleation on particles was also confirmed to accelerate the growth of pre-existing hydrogen micropores and cavities. Although continuous nucleation occurred together with the growth of pre-existing hydrogen micropores, the effects of the pre-existing hydrogen micropores, especially those located on grain boundaries, were predominant in the overall damage evolution. It seemed likely that supersaturated hydrogen in the aluminum alloys might also make an appreciable contribution to cavitation during high-temperature loading.

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### 1. Background

During the last decade, attempts have been made to introduce the quick plastic forming process (QPF) into the automotive industry [1]. QPF takes place at even higher forming rates and lower temperatures than superplastic forming (SPF), resulting in successful implementation of QPF as a cost-effective and higher-volume manufacturing alternative to SPF in the case of aluminum alloy components. The major mechanism of deformation in the case of SPF is grain boundary sliding (GBS) [2,3], while a com-

bined contribution of GBS and solute drag creep (SDC) is indispensable in the case of QPF [2,3], preserving a relatively large strain rate sensitivity parameter,  $m$ , and hence reasonable deformability.

Hot formability is, however, often limited by cavitation during forming, making cavity formation, growth and coalescence behavior an issue of primary importance in improving deformability [2–6]. It has been reported that cavities unidirectionally align along the tensile axis, exhibiting a string-like formation [2,7], under conditions in which SDC controls deformation; in contrast, during GBS, cavities coalesce perpendicularly to the tensile axis, with a more isotropic spatial distribution [2,7]. Possible cavitation mechanisms include: (i) GBS, leading to stress concentration at triple junction points and/or grain bound-

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ary ledges; (ii) cracking of particles, especially those located on grain boundaries; and (iii) vacancy condensation on grain boundaries [8,9]. Kassner and Pérez-Prado [10] reported that cavity nucleation only occurred on high-angle grain boundaries with grain boundary particles. It has also been frequently reported that transverse grain boundaries are common locations for cavitation [9]. However, it is still not well established by what mechanism and at which locations cavities nucleate.

High-resolution X-ray microtomography has revealed the existence of high-density micropores in most aluminum alloys, due to the precipitation of molecular hydrogen [11,12]. The hydrogen content in aluminum alloys is usually three to four orders of magnitude larger than the hydrogen solubility in solid aluminum alloys, due mainly to the existence of a hydrogen solubility gap at the melting temperature of aluminum and the presence of a surface oxide layer. It has been reported that supersaturated hydrogen in solid aluminum is predominantly partitioned to micropores [11]. The trap site occupancies of normal interstitial lattice sites, solute atoms, dislocations and grain boundaries are elevated with increases in hydrogen content [11]. The number density of micropores exceeds 10,000 per  $1 \text{ mm}^3$  in most wrought aluminum alloys [13], and it has been reported that the hydrogen micropores do not close even through subsequent extensive hot and cold plastic deformation [12,14]. Moreover, we have recently discovered [14,15] that such micropores exhibit premature growth under external loading at room temperature, thereby inducing ductile fracture. The well-known particle fracture mechanism operates only incidentally during the ductile fracture of aluminum alloys [14,15].

Given the recently revealed effects of micropores on room-temperature deformation, it is reasonable to assume that pre-existing hydrogen micropores may also contribute to damage evolution at high temperatures, at least to some extent. In the present study, synchrotron X-ray microtomography was applied to the in situ observation of deformation and fracture in Al–Mg alloys at a high temperature. Such high-resolution three-/four-dimensional (3-D/4-D, i.e. 3-D plus time) imaging readily enables the visualization of individual cavities throughout deformation, providing a unique opportunity to quantitatively analyze the initiation, growth and coalescence of each cavity. In addition to this visualization of cavities and particles, crystallographic grains were visualized by doping gallium into grain boundaries, thereby identifying the location where each cavity nucleated.

## 2. Experimental methods

### 2.1. Sample preparation

High- and low-purity 5086 alloys (hereinafter HP and LP, respectively) were obtained from Furukawa-Sky Aluminum Corp. Alloy LP had a chemical composition (in mass percent) of 4.5 Mg, 0.7 Mn, 0.12 Cr, 0.20 Fe, 0.15

Si and the balance Al. Alloy HP had a chemical composition of 4.5 Mg, 0.7 Mn, 0.13 Cr, 0.04 Fe, 0.04 Si and the balance Al, being characterized as a low Cr and Si alloy suitable for SPF. Another Al–Mg alloy, with reduced Mn and Cr content (hereinafter HP-C, as a coarse-grained high-purity alloy), was specifically prepared for identifying the location where each cavity nucleated. Alloy HP-C had a chemical composition of 4.5 Mg, 0.2 Mn, 0.0 Cr, 0.03 Fe, 0.02 Si and the balance Al, and was not compatible with the industrial standard of 5086 alloy due to its low Cr content. Almost all of the grains in alloys HP and LP had a grain size smaller than  $10 \mu\text{m}$ , whereas those of HP-C typically ranged between 50 and  $200 \mu\text{m}$ . Tensile specimens similar to those used in the previous study [14] ( $0.6$  (width)  $\times$   $0.6$  (thickness)  $\times$   $3$  (length) mm in gauge section) were machined from as-received  $1.3 \text{ mm}$  thick plates, using an electrodischarge machine wire eroder, with tensile load applied in the rolling direction.

### 2.2. Tomographic imaging

A high-resolution X-ray CT experiment was performed using the X-ray imaging beamline, BL20XU, of the SPring-8 facility. Samples were positioned in Experimental Hutch 1, which is located approximately  $80 \text{ m}$  from the X-ray source. A monochromatic X-ray beam, with a photon energy of  $20 \text{ keV}$ , generated by a liquid-nitrogen-cooled Si (111) double-crystal monochromator, was used. The image detector consisted of a cooled  $4000$  (H)  $\times$   $2624$  (V) element CCD camera (with an effective pixel size of  $5.9 \mu\text{m}$ , used in  $2 \times 2$  binning mode), a single-crystal  $\text{Lu}_2\text{SiO}_5\text{:Ce}$  scintillator and a  $\times 20$  lens. The image detector was positioned  $60 \text{ mm}$  behind the sample, thereby making the imaging system sensitive to phase modulation. In total,  $1500$  radiographs, scanning  $180^\circ$ , were obtained in  $0.12^\circ$  increments. The entire cross-section of the specimen and a region about  $551 \mu\text{m}$  high were captured on the CCD camera. Image slices were reconstructed from a series of projections based on the conventional filtered backprojection algorithm. An isotropic voxel with a  $0.42 \mu\text{m}$  edge was achieved in the reconstructed slices. The gray value in each dataset was calibrated such that the linear absorption coefficient of  $0\text{--}40 \text{ cm}^{-1}$  fell within an 8-bit grayscale range between 0 and 255.

In situ tensile tests were performed using a material test rig specially designed by the present authors for X-ray CT observation at high temperatures. An amorphous carbon tube was used as an axisymmetric load frame, resulting in negligible X-ray absorption. The test rig was equipped with upper and lower microring heaters ( $220 \text{ V}/340 \text{ W}$  each) to heat the tomographic specimens to  $923 \text{ K}$ . Thermal drift of a specimen could be suppressed to some extent by circulating water through channels machined in the upper and lower components. Tomographic scans were performed at  $773 \text{ K}$ , at successive increments of applied cross-head displacement, while holding a sample at fixed cross-head displacements. Approximate strain rate during loading was

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