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論 文 名: Internal hydrogen partitioning and related embrittlement behavior in Al-Zn-Mg-Cu aluminum alloys (Al-Zn-Mg-Cu アルミニウム合金における水素分配と脆化挙動)

区 分 :甲

論文内容の要旨

The main objective of this thesis is to understand the hydrogen partitioning and related embrittlement behavior in Al-Zn-Mg-Cu aluminum alloys. For this purpose, a series of investigations such as the 3D-image-based analyses of fracture behavior and hydrogen repartitioning during deformation are performed. The thesis consists of six chapters as follows:

Chapter 1 describes a general introduction and related background of the present research. The current understanding of hydrogen embrittlement is that hydrogen trapped at various trap sites such as dislocations and grain boundaries results in premature fracture at low applied strains. Although a number of experimental, numerical and theoretical approaches have been reported, the current literature does not often provide reasonable interpretations for observed hydrogen embrittlement behavior. In a recent attempt, it has however been revealed with the help of a first principles simulation that an unrealistically high hydrogen concentration at interstitial lattice is necessary for hydrogen embrittlement process. Based on such a finding, this thesis focusses on the in-situ observation of hydrogen embrittlement behavior and associated estimation of hydrogen repartitioning among various trap sites. High-Zn Al-Zn-Mg-Cu aluminum alloys have been used as a model material. This is because further improvement in mechanical properties is expected if high susceptibility for hydrogen embrittlement is overcome in this alloy system.

Chapter 2 reveals the general 3D microstructural features of the present high-Zn Al-Zn-Mg-Cu aluminum alloys. Various hydrogen trap sites have been observed in 3D, together with their morphological information. Hydrogen micropores, which are one of the major hydrogen trap sites, are heterogeneously nucleated on the intermetallic particles. The intermetallic particles, which are also possible hydrogen trap sites, affect hydrogen precipitation behavior through heterogeneous nucleation of hydrogen micropores on intermetallic particles.

Chapter 3 reports the general fracture behavior of the Al-Zn-Mg-Cu aluminum alloys. Fe and Si contents have been widely varied between 0.01 and 0.30 % in order to examine the effects of Fe- and Si-bearing intermetallic particles. Quasi-cleavage cracks are initiated at relatively low applied strain levels. It has been revealed that the fractional area of quasi-cleavage cracks on fracture surface increases with the decrease in Fe and Si contents. It has been generally accepted that dislocations are the hydrogen trap site associated with quasi-cleavage fracture. It is therefore inferred that hydrogen accumulation along dislocations is affected by the existence of intermetallic particles, probably due to predominant hydrogen trapping at the particles. In

addition, damage behavior of various intermetallic particles such as Al₇Cu₂Fe and Mg₂Si has been characterized by means of the 3D image analysis technique called microstructural tracking. It has been revealed that the fracture of Al₇Cu₂Fe particles has a dominant influence on the ordinary ductile fracture in the high-Zn Al-Zn-Mg-Cu aluminum alloys, and that premature fracture from hydrogen micropores is limited to the vicinity of cracks. It can be concluded that the ordinary ductile fracture originated from the cracking of Al₇Cu₂Fe particles competes directly with the initiation of quasi-cleavage cracks due to in-situ hydrogen accumulation during deformation. It is therefore important to characterize the in-situ deformation behavior of this alloy, which is then mentioned later in chapter 4.

Chapter 4 illustrates the behavior of dislocations in terms of microscopic strain distribution. The microstructural tracking technique has been used to obtain 3D strain distributions. Hydrogen induced strain localization has been visualized in 3D by tracking densely distributed intermetallic particles. The strain localization has been observed as a form of obliquely aligned shear bands. It can be seen that strain localization is enhanced with the increase of holding time at each loading step, indicating that more hydrogen is repartitioned to strain localization regions while holding time. In addition, it has been found that a great number of nano voids are observed after deformation through the precise analysis of the origins of measured hydrostatic tension. Direct observation of nano voids has successfully been performed by employing the state-of-the-art ultra-high resolution X-ray tomography technique combined with the high angle annular dark field (i.e., HAADF) scanning transmission electron microscopy imaging technique. It is natural to assume that nano voids can potentially serve a dual role as a fracture origin and a hydrogen trap site. No evidence for hydrogen embrittlement originated from nano voids has however been observed in a series of ultra-high resolution 3D images. Instead, it can be assumed that the majority of hydrogen is repartitioned to nano voids in strain localization regions during deformation due to their high density. A hydrogen embrittlement model has been finally proposed on the basis of these findings, in which in-situ hydrogen repartitioning necessary for hydrogen embrittlement to occur is considered.

Chapter 5 estimates hydrogen repartitioning behavior among various trap sites during loading, which is necessary for establishing a mechanical criterion for hydrogen embrittlement to occur. Vacancy production and dislocation multiplication due to plastic deformation have been estimated to precisely estimate the density values for all the hydrogen trap sites. The hydrogen repartitioning analysis has revealed that estimated hydrogen concentration at dislocations is associated with the initiation of a quasi-cleavage crack, and a critical hydrogen concentration is around $2.5 \times 10^{17} \text{ m}^{-3}$. The hydrogen embrittlement model proposed in chapter 4 has been modified on the basis of the findings obtained in chapter 5. The hydrogen trap density together with the in-situ increase in vacancy, nano voids and dislocations. It is of crucial importance that hydrogen repartitioning behavior is controlled by changing the content and species of intermetallic particles thereby controlling the resistance for hydrogen embrittlement.

Chapter 6 summarizes all the results and discussions of the present studies, addressing some of the possible future directions by extending the ideas explored here. A consistent theme throughout the thesis work was the emphasis on understanding realistic microstructural features and complex fracture behavior. The new concept for the mechanical understanding of hydrogen embrittlement behavior proposed in the present thesis can be utilized for the various works such as the practical control of hydrogen embrittlement and the mechanical modelling and/or simulation of a hydrogen-assisted fracture.