DOE/ER/45255--4

DE92 018714

ENVIRONMENT-INDUCED EMBRITTLEMENT:

1) Stress Corrosion Cracking and Metal-Induced Embrittlement
2) Environmental Embrittlement of Iron Aluminide Alloys

Final Report

L. A. Heldt
W. W. Milligan
C. L. White

Department of Metallurgical and Materials Engineering
Michigan Technological University
Houghton, Michigan 49931

September 1, 1986-August 31, 1991

PREPARED FOR THE
U.S. DEPARTMENT OF ENERGY
AGGREEMENT FG02-86ER 45255

ABSTRACT

This research program has included two thrusts. The first addressed environment-induced embrittlement in a parallel study of stress corrosion cracking and metal-induced embrittlement. This work has examined (1) mechanical properties as influenced by embrittling environments, (2) fractography and crystallography or transgranular cracking, (3) the mechanics of cracking, (4) the extent and role of local plastic flow, and (5) local chemistry within stress corrosion and metal-induced cracks.

The embrittlement of iron aluminide alloys by air was addressed by determining the effect of water and hydrogen upon the mechanical properties. Slow strain rate testing in aqueous environments was carried out at controlled anodic and cathodic potentials. The effect of cathodically charged hydrogen and the effect of subsequent baking were measured. Environmental susceptibility was measured as affected by alloy composition, microstructure and degree of ordering.

I. Parallel Studies of Stress Corrosion Cracking (SCC) and Metal-Induced Embrittlement (MIE)

The primary model system employed in this work was beta brass embrittled by water (for SCC) and by gallium (for MIE). Methods were developed to grow large single crystals from which the test specsimens were prepared. Alpha brass was employed for certain of the studies.

A. Stress Corrosion Cracking

Crystallography and Mechanics of Transgranular SCC

The crystallography of cracking was determined for the single crystals by light goniometry techniques. Cleavage-like fracture occurs on (100) planes in 110 directions. The fracture stress depends strongly upon crystallographic orientation. A large number of crystals having various orientations were subjected to slow strain rate testing in the embrittling water environment. It was found that for crystal combinations of strain rate and electrochemical potential the fracture stress resolved normal to the fracture plane (100) was nearly constant. This observation of a critical resolved normal stress was investigated further; it was found that for a given electrochemical potential the strain rate which provided for CRNS behavior also produced a minimum fracture stress. CRNS behavior is characteristic of several materials exhibiting intrinsic cleavage and is known as Sohncke's Law. This work has, accordingly, demonstrated that transgranular SCC in this system (and, it is believed, in general) results in a cleavage-like fracture appearance and is driven, macroscopically in the same manner as intrinsic cleavage.

Accompanying Plastic Processes

The apparent brittle behavior is, however, accompanied by considerable local plastic activity, which has an important role in the embrittlement process. The large beta brass crystals afford an excellent opportunity for measuring the extent of flow in the vicinity of a propagated crack by X-ray rocking curve analyses. By this means it was determined that a great deal of deformation occurs in the vicinity of the propagating crack and that it is restricted to $\gtrsim 50\mu m$ depth.

This finding suggested a study of the effect of the embrittling environment on near-surface deformation, in which X-ray diffraction was employed to measure microstrain on the surfaces of brass samples strained in air and in an embrittling environment of ammonia solution. It was found that microstrains accumulate much more rapidly during straining in the embrittling environment. These experiments were followed by a systematic study of prestrain effects on subsequent straining in the SCC environment. A remarkably regular behavior was found for the prestrain reducing the subsequent SCC ductility. Straining in the SCC einvironment is about three times as effective (per increment of strain) in bringing the material to fracture as is straining in air. However, with the addition of an SCC inhibitor to the environment, the straining effect was equivalent to that in air.

B. Metal-Induced Embrittlement

The gallium-induced embrittlement of beta brass was characterized by Auger spectrometric studies of the leading edge of fracture and by mechanical response.

Surface Chemical Analysis

Scanning Auger analysis of the fracture surface containing a gallium-starved crack front reveal that Ga is present on the cleavage-like fracture, but not in a subsequent ductile overload region. The Ga is most highly concentrated along the fracture steps; but, importantly, only about one monolayer of coverage is sufficient to sustain propagation of the crack.

Discontinuous Crack Propagation in Metal-Induced Embrittlement

Detailed studies of crack propagation have provided the first clear evidence for discontinuous crack propagation to be reported in the

Western literature and the first fractographic evidence to be reported anywhere. Crack jumps of tens to hundreds of microns were measured. The time interval between jumps may be required to generate a critical amount of crack tip strain and/or may be required for a chemical process of Ga diffusion and reaction. Varying the crosshead velocity had little effect on the crack jump distance or the magnitude of crosshead displacement between jumps, indicating that crack advance depends more upon the magnitude of crosshead displacement rather than the time per se between events -- thus implying a mechanical process is rate controlling rather than a chemical one. This would substantiate a model postulated by Popovich and others.

II. Environmental Embrittlement of Iron Aluminide Alloys

These alloys are candidate materials for corrosion resistant service. The impetus for this work was the report by workers at Oak Ridge National Laboratory (Liu, Lee and McKamey) that the low ductility generally measured for iron aluminides strained in air is the result of environmental embrittlement, probably by water vapor. Testing in vacuum or dry oxygen results in a significant increase in tensile ductility.

Tensile Properties in Controlled Environments

In order to determine the effects of water vapor in air, the testing environment was controlled at various levels of relative humidity. A strong sensitivity was seen: a large ductility loss occurring at less than 10% RH with further, smaller increments to higher humidity levels. Vacuum environments provide for ductilities which exceed those measured in desiccated air and these are, in turn, surpassed in an oxygen environment.

For testing in aqueous environments, it was seen that at open circuit electrochemical potential, the tensile properties are much the same as in air. Application of cathodic potentials (in the hydrogen reduction range) causes further ductility loss but anodic potentials do not enhance ductility beyond the value measured in air. Thus, the embrittlement process appears to be caused by or is assisted by cathodic hydrogen, but driving the hydrogen fugacity down by in situ anodic polarization does not have a converse effect.

Following these observations, alloys were cathodically precharged in acid solutions before testing in air or vacuum. This caused severe embrittlement, thus demonstrating that internal hydrogen is capable of causing the embrittlement. This was confirmed by restoring full ductility to the material by baking treatments. Room temperature storage in vacuum does not remove the hydrogen, which suggests a very low diffusivity at that temperature. In contrast to cathodic charging, preexposure of samples to water vapor saturated air for up to 42 days had no effect; such treatments do cause hydrogen embrittlement of AlZnMg alloys.

The role of the environment appears to be of significance in crack propagation rather than crack initiation. For tests in nonembrittling environments profuse small cracks are observed; in the water bearing environments, these propagate readily at low stress intensities.

Effects of Alloy Composition, Structure Substructure on Susceptibility

Systematic measurements of environment-induced embrittlement have been made of iron aluminides containing 15 to 40 a/o aluminum. It was found that the ductility enhancement provided by dry environments was considerably larger for 28 to 35% Al than at lower concentrations. However, >38% Al alloys were very brittle in all environments, apparently because of fragile grain boundaries.

X-ray diffraction and electron microscopy studies revealed that the increased ductility observed in dry environments coincides with an increase in order for the 28-35% Al alloys. For very low (<18%) Al concentrations the structure is disordered and large ductilities are observed for dry oxygen. Alloys containing 23-27% Al are brittle in all environments -- the result of a substructure containing both ordered and disordered phases.

It is seen that alloy composition and structure are important factors in control of the intrinsic ductility, which can only be measured in dry environments. Resistance to environmental embrittlement may be addressed by alloying additions or by protective coatings.

Publications Resulting from this Research

"Interface Segregation and Cohesion," C. L. White in Diffusion in High Technology Materials, ed. by G. Gupta, A. D. Romig and M. A. Dayanada, Trans. Tech., 1988.

"The SCC of Single Crystals of Beta Brass in Water: Fractography and Crystallography," W. K. Blanchard Jr., Met. Trans. A, 20, pp. 1439-1444 (1989).

"Environmental Effects on the Cracking of Engineering Materials," D. B. Kasul and L. A. Heldt, Materials Research Society Bulletin, 14, pp. 37-43 (1989).

"Relationships Between Plasticity and Stress Corrosion Cracking," D. B. Kasul, C. L. White and L. A. Heldt in Environment-Induced Cracking of Metals, ed. by R. P. Gangloff and M. B. Ives, National Association of Corrosion Engineers, 1989 (reprints enclosed).

"An Auger Spectrometric Study of the Crack Tip Surface Chemistry for Liquid Metal Embrittlement: Beta Brass Embrittled by Gallium," P. H. AuYeung, J. T. Lukowski, L. A. Heldt and C. L. White, Scripta Metall. Mater., 24, pp. 95-100 (1990) (reprints enclosed).

"Discontinuous Crack Propagation in Gallium-Induced Liquid Metal Embrittlement of Beta Brass," J. T. Lukowski, D. B. Kasul, L. A.Heldt and C. L. White, Scripta Metall. Mater., 24, pp. 1959-1964 (1990) (reprints enclosed).

"Environmental Embrittlement of an Fe-Al Alloy," D. B. Kasul and L. A. Heldt, Environmental Effects on Engineering Materials, ed. by R. H. Jones and R. E. Ricker, The Minerals, Metals and Materials Society, 1991 (reprints enclosed).

"Effect of Environment on the Mechanical Properties of an Fe-24.6Al Alloy," D. B. Kasul and L. A. Heldt, Scripta Metall. Mater., 25, pp. 1047-1051 (1991) (reprints enclosed).

"Effects of Alloy Composition on Environmental Embrittlement of B_2 Ordered Iron Aluminides," R. J. Lynch, L. A. Heldt and W. W. Milligan, Scripta Metall. Mater., 25, pp. 2147-2151 (1991) (reprints enclosed).

DATE
FILMED
8/31/92