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Alloy Development for Irradiation Performance

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CONTENTS

The HFIR-CTR-39, -40, and -41 experiments contain rod tensile specimens of the nickel-doped 9 Cr-1 MoVNb and 12 Cr-1 MoVW alloys and the 2 1/4 Cr-1 Mo alloy. Different nickel levels result in different helium levels generated during irradiation. Testing of these alloys will provide data on the effect of helium on tensile properties. Irradiations will be at 300, 400, and 500°C to a midplane fluence equivalent to 12 dpa.

The Path A PCA with various pretreatments and 20%-cold-worked type 316 stainless steel (CW 316) were irradiated in HFIR at 300 to 600°C to fluences producing up to 9.7 dpa and 620 at. ppm He. Very tittle melting was observed at 500°C and below, but swelling at 600°C ranged from 0.2 to 1.26. Swelling appears coupled to precipitate phase and dislocation microstructural development.

Path A PCA in several preirradiation grain boundary microstructural conditions was irradiated in HFIR at 300 to 600°C to 10 dpa, together with 20%-cold-worked (N lot) type 316 stainless steel (CW 316). None of the samples show significant grain boundary bubble formation at 300 to 500°C. At 600°C, PCA-B1 and -B2, with stable, moderately sized grain boundary MC, have the finest dispersions of grain boundary bubbles.

Strain controlled fatigue tests were performed in vacuum at 650°C on 20%-cold-worked type 316 stainless steel irradiated in the HFIR at 550°C. Fluences ranged from 0.85 to 1.4 x 10^{26} neutrons/m^2. Little effect of irradiation was observed, but testing at 650°C failed to show the 10^7-cycle endurance limit observed at 550°C.

Different techniques for consolidation of rapidly solidified alloys which are available or are under study at the present time include conventional consolidation techniques (hot extrusion, HIP, ..), high velocity consolidation of atomized partially solidified particulates and dynamic powder compaction (DPC). This report describes the results of dynamic compaction of Path A alloy with increased carbon and titaniwn content. The microstructure of the as-compacted alloy is highly complex, evidencing an extreme degree of deformation. TEM revealed very high dislocation and twin density reflecting high hardness of the as-compacted alloy. Annealing studies revealed that recovery and recrystallization processes in dynamically compacted alloy are slower than in conventionally treated materials. High dislocation density appears to be an intrinsic property of the dynamic compaction process and it may be potentially useful in developing materials for irradiation performance. Other potential applications of dynamic compaction include preparation of graded materials and ceramic materials.

An oxide dispersion stabilized 316 stainless steel has been prepared by mechanical alloying of a few 100 Å size yttria powders with attrited flakes of 316 staintess steet prealloyed with 1 wt% aluminum. Alloys with 4 vol% and 5 vol% yttria additions have been prepared and they show significant improvements in microstructural stabitity. Austenitic stainless steel containing 4 vol% yttria showed very high room temperature YS and UTS, with sufficient ductility. Stress rupture testing at 650°C as a function of different thewnomechanicat treatments is currently in progress.

Samples of solution annealed types 316 and 347 stainless steel (SA 316 and SA 347) and 20%-cold-worked type 316 stainless steel (CW 316) have been irradiated in the High Flux Isotope Reactor (HFIR) at 55°C. No voids or bubbles were detected. All samples contained a fine "black spot" component of the dislocation structure. Both SA 316 and SA 347 contained a high concentration of Frank interstitial loops (14–30 nm diam) but no dislocation networks, whereas CW 316 shows network recovery with few Frank loops.

Previously it was reported that a 30,000 lb heat of a 12Cr-1Mo steel melted to the specification of Sandvik HT-9 was procured by General Atomic Company from Electralloy Corporation for the Ferritic Steel Program. This heat of argon-oxygen decarburization (AOD) 12Cr-1Mo steel has, since, been electroslag remelted (ESR) by Universal Cyclops Corporation in Pittsburg, and converted to plate and bar stock. This remelted heat (No.9607R2) was within the chemical specification requirement of HT-9. The remelted ingot was homogenized at 1100C for 16 hrs prior to conversion to the product forms. After conversion to plate and bar stock, each form was given a stress relief at 700C for 6 hrs. Subsequent mechanical property analysis on the 0.625 in (1.58 cm) plate has shown that it meets the strength and tensile elongation specifications. The UTS was 121 ksi (835 MPa), the yield stress was 87.5 ksi (604 MPa), and the elongation was 21%. The upper shelf energy determined on standard Charpy impact specimens was 85 ft-lbs (115J) as compared to the upper shelf of the AOD heat of 66 ft-lbs (89J). Microstructural examination showed a uniform tempered martensitic structure with a delta ferrite content less than 1%.

To be reported in the next semiannual report.

The four distinct HAZ microstructures which were identified in HT9 GTA welds were simulated in the Gleeble in order to produce bulk microstructural Charpy V-notch samples suitable for evaluation of notch toughness. Samples both transverse and longitudinal to the rolling direction (R.D.) of AOD melt plate stock were evaluated. Following the Gleeble thermal treatment the samples were given a postweld heat treatment (PWHT) for 1 hour at 760°C. Charpy tests of the individual microstructures were performed over the temperature range from -60 to 200°C. The toughness values of samples oriented transverse to the rolling direction (notch aligned parallel to R.D.) fell in a wide scatterband which enclosed the base metal toughness at low temperatures and was slightly less than the base metal value on the upper shelf. When samples oriented parallel to the R.D. (notch transverse to R.D.) were evaluated the toughness of three of the microstructural regions was again equivalent to that of the base metal.

Tensile tests were performed to determine the effect of hydrogen on the ductility of a quenched-and-tempered 12Cr-1Mo alloy from the National Fusion Heat-ESR processed. specimens were cathodically charged with hydrogen at 0.003 A/cm^2 and 0.006 A/cm^2 for 15 to 150 minutes. In the uncharged condition, the ESR specimens failed by intergranular rupture with prominent secondary cracking along prior austenite grain boundaries. For the least severe cathodic charge (0.003 A/cm^2 – 15 minutes), the tensile ductility (R_A) decreased 33% and the degree of secondary cracking increased. At 0.003 A/cm^2 for 150 minutes, the ductility decreased further and the fracture mode changed to classical intergranular fracture. These results generally mztched those for the AOD processed 12Cr-1Mo alloy that had been tested previously.

Auger analysis indicates that there is segregation of phosphorous and the carbide-forming elements Cr, Mo, W to the prior austenite grain boundaries. In the literature, phosphorous has been shown to embrittle these boundaries, especially in the presence of hydrogen.

The combination of tensile data on hydrogen charged ESR and AOD 12Cr-1Mo steet present several reasons for concern regarding the susceptibility of this alloy to hydrogen embrittlement. Earlier results of hydrogen charged Sandvik HT-9 are addressed in terms of these more recent findings.

In addition to the ferritic steels previously tested, experiment HFIR-CTR-33 also contained tensile specimens of 20%-cold-worked type 316 stainless steel for irmdiation at about 55°C. The tensile behavior of the type 316 stainless steel was determined at room temperature and 300°C, and the results showed the irmdiated properties of the two classes of steels to be quite similar.

Tensile tests at 700°C a nickel-doped 9 Cr-1 MoVNb and 12 Cr-1 MoVW steels that were irmdiated at 55°C in HFIR to produce up to about 50 at. ppm He indicated that there was no helium embritttement. These results contrast with literature data showing that very small amounts of helium can cause a large decrease in ductility for many alloys when tested under equivalent conditions.