AN APFIM AND TEM STUDY OF Ni_4Mo PRECIPITATION IN A COMMERCIAL Ni-28% Mo-1.4% Fe-0.4% Cr wt. % ALLOY

CONF-980713-1 RECEIVED

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MAR 06 1998

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Ni-Mo alloys containing at least 26 wt.% Mo have a negligible corrosion rate in boiling 10% hydrochloric acid and are therefore used in corrosive environments. A series of commercial Ni-Mo alloys has been developed with subtle variations in chemical composition. These alloys usually contain ~28 wt.% Mo with additions of up to 5 % Fe and Cr. A significant amount of research has been performed to understand the microstructure and properties of these alloys, although most of the effort has concentrated on the Ni-Mo binary system.¹ In some alloys with low Fe and Cr contents, a severe embrittlement problem has been observed due to the formation of the Ni_4Mo (D_{14} -ordered) phase within the microstructure. The calculated section of the ternary Ni-Mo-Fe phase diagram in Fig. 1 illustrates that the addition of Fe stabilizes Ni_3Mo with respect to the brittle Ni_4Mo phase.

This research focuses on a commercial alloy with nominal composition Ni-28% Mo-1.4% Fe-0.4% Cr-0.1% Mn-0.003 wt. % C. The material supplied was a heat treatment coupon which had been attached to a large vessel during fabrication. Assessment of the chemical analysis of the alloy suggested that detrimental phases could be present or might appear during subsequent repair work. Therefore, it was important to assess the microstructural condition of the vessel, and in particular the kinetics of precipitation of Ni_4Mo .

Transmission electron microscopy has established that following a solution annealing treatment at 1066°C and water quenching, the alloy is single phase, although there is some evidence for short range ordering (DO_{22} -ordered) in the selected area diffraction pattern (Fig. 2a).² Statistical analysis of atom probe data from the as-quenched specimen, using both the Johnson and Klotz and other Markov chain techniques, confirmed the presence of short range order. This material has a hardness of 196 ± 5 HV. The alloy has been examined after subsequent tempering at a number of temperatures including 700°C and 750°C for 0.5, 1, 2, 10, and 100 h. The corresponding hardnesses at 700°C were 204, 212, 260, 352 and 384 ± 5 HV and 211, 232, 327, 392 and 402 ± 5 HV at 750°C. The increase in hardness is due to the precipitation of Ni_4Mo (Fig. 2b); the kinetics of precipitation described by the hardness values follow a C curve. A significant decrease in both toughness and ductility was also observed as tempering progressed.

Atom probe investigations have observed small (5-10 nm) platelets of ordered Ni_4Mo within the matrix phase after tempering for 0.5h at 750°C (Fig. 3a) which progressively increase in size as further tempering proceeds (Fig. 3b). The concentration of Fe and Cr was found to be significantly higher in the matrix than in the Ni_4Mo precipitates (Fig. 4) and the partitioning increased with tempering time. Furthermore, atom probe composition profiles (Fig. 5) showed that there was significant depletion of Fe in the Ni_4Mo and a corresponding enrichment in the matrix adjacent to the interface. This behavior indicates an aversion of Ni_4Mo for Fe, and concurs with the suppression of the embrittling Ni_4Mo phase in alloys with more than 5 % Fe.³

1. C.R. Brooks, J.E. Spruiell and E.E. Stansbury, *International Metals Reviews*, 29 (1984) 210.
2. H.M. Tawancy, *Journal of Materials Science*, 30 (1995) 522.
3. This research was sponsored by the Division of Materials Sciences, U. S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp and through the SHaRE Program under contract DE-AC05-76OR00033 with Oak Ridge Associated Universities. This research was conducted utilizing the Shared Research Equipment (SHaRE) User Program facilities at Oak Ridge National Laboratory.

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FIG. 1 - Calculated section of ternary Ni-Mo-Fe system.

FIG. 2 - Selected area electron diffraction patterns [001] showing reflections attributable to (a) DO_{22} ordering in the as-quenched material and (b) $D1_3$ ordering in material tempered for 10 h at 750°C.

FIG. 3 - Field ion micrographs showing (a) the early stages of precipitation and (b) a boundary between a larger $D1_3$ -ordered precipitate and the matrix.

FIG. 4 - The partitioning of Fe and Cr between Ni_4Mo and the matrix with tempering time at 750°C.

FIG. 5 - An atom probe composition profile illustrating the diffusion of Fe in the vicinity of the transformation interface.

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M98005021

Report Number (14) ORNL/CP--96510
CONF-980713--

Publ. Date (11) 199802
Sponsor Code (18) DOE/ER, XF
UC Category (19) UC-400, DOE/ER

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