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APPLICATION OF ATOM PROBE MICROANALYSIS FOR UNDERSTANDING MICROSTRUCTURE EVOLUTION IN NICKEL BASE SUPERALLOY WELDS

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The characterization of the microstructure evolution during welding of nickel base superalloys is required for efficient reuse and reclamation of used and failed components.[1] Previous atom probe analysis of electron-beam and laser-beam welds revealed complex alloying elemental partitioning between the γ and γ′ phases.[2] Rapid cooling conditions in the weld leads to non-equilibrium partitioning and large amplitude Cr and Co levels in the γ phase. These results indicated that there is a strong relationship between weld cooling rate and the precipitation of γ′ precipitates from the γ phase. To understand and develop predictive models, a systematic investigation of the microstructure evolution in CM247DS alloy under controlled thermomechanical conditions are being performed. This paper describes some recent results on the elemental partitioning between γ and γ′ phases obtained with atom probe microanalysis.

Samples of directionally solidified CM247DS alloy (Ni – 8 wt. % Cr – 9% Co – 5.5% Al – 0.8% Ti – 0.1% Nb – 0.6% Mo – 3.2% Ta – 9.5% W – 0.08% C) were machined from the castings. The samples were solutionized at 1300 °C for 5 minutes and were cooled at 0.17 to 75 °C s^{-1} in a Gleeble® thermomechanical simulator. In addition, some samples were water-quenched from 1300 °C. Microstructural analyses were performed on these samples by transmission electron microscopy (TEM), atom probe field ion microscopy (APFIM) and atom probe tomography (APT).[3,4]

The microstructures obtained at different cooling rates are compared in Fig. 1. The samples cooled at a rate of 0.17 °C s^{-1} [Fig. 1(*a*)] from 1300 °C consists of coarse cuboidal γ′ precipitates. At a faster cooling rate of 75 °C s^{-1}, the γ′ precipitate size decreased [Fig. 1(*b*)] and the number density increased. In addition, the shapes of the γ′ precipitates tended to exhibit irregular shapes instead of the normal cuboidal shape. In the water-quenched condition, the number density of the γ′ precipitates increased and their size decreased [Fig. 1(*c*)]. In addition, the shapes of the precipitates became more spherical. Some of the γ′ precipitates were found to be interconnected, as shown by the arrows in Fig. 1(*c*). APT analyses of the water-quenched samples was performed to measure the partitioning characteristics as well as the γ′ morphology. The atom map (Fig. 2) shows complex γ′ morphology and indicates no regular cuboidal or spherical morphologies. Some of the γ′ particles exhibited both flat and curved interfaces with the γ phase. Composition profiles were measured from these data and are shown in Fig. 2(*b*). The composition profiles indicate a Cr-enriched γ regions and Al-enriched γ′ regions. Small Co-enriched regions were observed within the γ′ phase. Additional analyses showed that W and Ti did not partition exclusively to either the γ or γ′ phases. This result was consistent with one-dimensional APFIM analyses. In addition, an Al-enriched region was observed within the γ phase. The 15 at. % Cr iso-concentration surface delineating the γ-γ′ interface at region A reveals that the γ phase is continuous along the neighboring γ′ precipitates and that the γ′ precipitates on either side of the γ phase ribbon in A were connected to each other. This interconnected morphology was also observed in TEM. In contrast, the γ phase in region B is continuous and the γ′ phases on either side appear to be separate precipitates.

These complex partitioning characteristics and changes in γ′ morphology need to be understood in terms of phase transformation mechanisms as well as implications of these characteristics on the expected mechanical properties. These results will be used to develop thermodynamic and kinetic models to predict microstructure evolution during welding.[5]

References

1. S. A. David *et al*., *Sci. Tech. Weld. Join.* **2** (1997) 79.
2. S. S. Babu *et al*., *Microscopy and Microanalysis* **4** (1998) 94.
3. M. K. Miller *et al*., Atom Probe field Ion Microscopy, Oxford University Press, 1996.
4. M. K. Miller Atom Probe Tomography: Analysis at atomic level, Kluwer Academic / Plenum Publishers New York, 2000.
5. Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by the Division of Materials Sciences, U.S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

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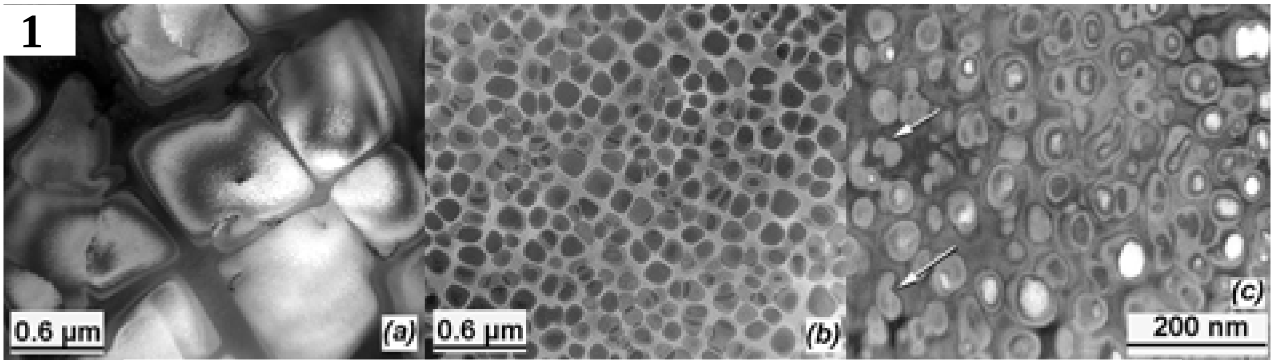

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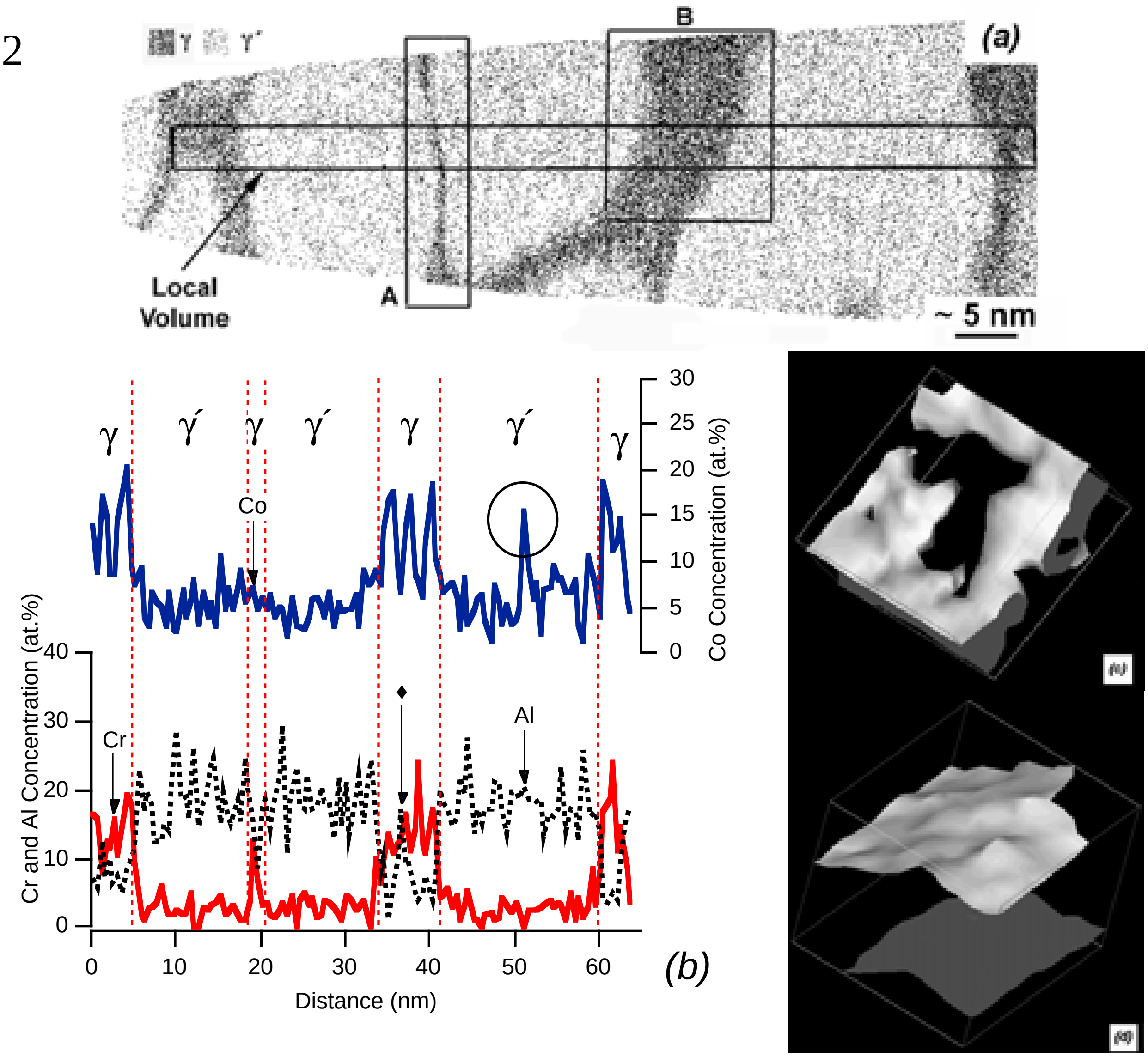

FIG. 1. Transmission electron micrographs (in [001] zone axis condition) of CM247DS alloy subjected to solutionizing at 1300°C for 5 min and cooled at different rates. Microstructures obtained at (*a*) 0.17 °Cs^{-1}, (*b*) 75 °Cs^{-1}, and (*c*) in water-quenched condition showing fine γ´ precipitates and complex morphology.

FIG. 2. APT analysis from CM247DS sample in the water-quenched condition. (*a*) Cr-atom image, (*b*) composition profiles obtained from the local volume region and iso-concentration plots (15 at. % Cr) from (*c*) regions A and (*d*) B.