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Research Involving Steel

The early part of this grant involved several projects relating to three steels, AF1410, an ultra-high strength, high toughness steel originally developed for carrier-based aircraft and in the space shuttle; Fe9Cr1Mo+(V, Nb), an advanced steel used in power generating equipment; and Fe2.25Cr1Mo, a common steel used in power generation.

Anomalous small angle X-ray scattering was used to follow the coarsening of Cr23C6 carbides in Fe9Cr1Mo+(V, Nb) steel exposed to prolonged periods of time at elevated temperatures. A graduate student supported in the grant previous to this one the microstructural changes associated with the rather rapid softening observed in this steel when it is fatigued at elevated temperatures. Part of the initial strength of the steel is derived from its various carbides. However at the initiation of this project, currently available SAXS (and SANS) cameras were not capable of resolving the larger Cr23C6 carbides and so it was not clear how the various carbides present were changing during high temperature fatigue. Anomalous small angle X-ray scattering (ASAX) offers the possibility of separating out the scattering caused by a particular scattering entity from the total scattering in a complex material containing a number of scattering systems. Thus it would be possible to follow the influence of various forms of high temperature service on the different carbides or other precipitates in an alloy. With the use of a synchrotron for the source of radiation a monochromatic beam of X-rays is obtained that can be tuned over a wide range. The phenomenon of anomalous scattering then is used to separate out the contributions to the total scattering from the various scattering species. To carry out the ASAXS measurements we constructed, working in cooperation with Dr. G. Long of NIST, a superb small-angle X-ray scattering camera. The experiments were performed at NSLS, Brookhaven National Laboratory.

The size distribution and volume fraction of the Cr23C6 precipitates were isolated from the distributions of all other precipitates by the ASAXS technique. Three X-ray wavelengths near the Cr K absorption edge were used to vary the scattering contrast of Cr23C6 while that of the other precipitates was left unchanged. Size distributions calculated from each scattering curve using a maximum entropy method were combined by a scattering contrast gradient analysis to isolate the volume-fraction size distribution of the chromium carbides. As a demonstration of the technique, behavior of these carbides was studied as a function of isothermal aging temperature. Mean diameter is smallest and Cr23C6 number density is highest after aging at 811 K. Above 811K, the mean diameter of the chromium carbides increases with increasing temperature.

SANS was used to follow the earliest stages of precipitation and growth of M2C carbides in the AF1410 steel. These carbides help to give the steel its high strength and toughness, so it is of interest to follow their nucleation and growth in the search for

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optimum composition and heat treatment. A large number of very small M₂C carbides is desirable. The SANS measurements were carried out by Dr. Andrew Allen, a visiting scientist from Harwell, England. They were complemented by a TEM investigation by Dr. Didier Gavillet, a visiting scientist from the Paul Scherrer Institute, Switzerland. Both were partially supported on the DOE project. The SANS and TEM results were combined with APFIM and theoretical studies carried out by other members of the faculty. Precipitation was studied as a function of isothermal aging time of the main secondary hardening M₂C phase. The results obtained by SANS and TEM were compared with the APFIM investigations and the theoretical models. The combined use of these techniques provided valuable diagnostic data for quantifying the development of microstructure in this class of steels. It was found that the observed precipitation behavior is consistent with the theory of precipitation at high supersaturations, but includes a surprising secondary burst of nucleation at precipitation half completion.

Work on the previous DOE grant on the elevated temperature fatigue behavior of the Fe₉Cr₁Mo+(V,Nb) steel had shown that tensile holds are more damaging than compressive holds (as would be expected) if the tests are carried out in vacuum. However the reverse is true if the tests are done in air. An explanation for this reversal had been given by McIvley, based on the effect of the oxide film grown during high temperature air tests. Graduate student Rena Hecht studied the effect in Fe_{2.25}Cr₁Mo, concentrating on the behavior of the oxide film, the mechanism by which the film causes damage, and its influence on fatigue life. Total strain control fatigue tests with a 120-second hold period at either peak compressive or tensile strain were conducted on annealed samples of the Fe_{2.25}Cr₁Mo steel. Tests were performed at the total strain range of 1% at 500C or 600C in air, 1.3 Pa or 0.0013 Pa vacuum. The nature of the hold and the environment affect fatigue life and surface crack patterns. Long, straight cracks perpendicular to the stress axis were seen on the oxidized surface of the specimens cycled with a compressive hold. The cracks in the oxide are periodically spaced. They resemble cracks observed in a brittle film on a ductile substrate after a tension test of the substrate. They also resemble the parallel multiple fractures that occur in a brittle matrix of a composite with ductile fibers undergoing tension. Both the model of a brittle film on a ductile substrate and of a brittle matrix composite were used to explain the observed intercrack spacing. Cracks in the oxide film lead to localized oxidation of the metal in the region around their intersection with the oxide-metal interface. These cracks penetrate the metal. Stress concentrations from deep grooves that form during compression hold fatigue, together with crack initiation from the oxide, lead to a shortened cycle life. The oxide film formed during a tensile hold tends to spall off at the next excursion into compression and thus is removed as a potential cause for harm.

Research Involving Nanocrystalline Metals

The synthesis, structure, and mechanical behavior of a number of nanocrystalline metals were investigated over the latter part of the grant. Synthesis was primarily carried out by inert gas condensation and compaction in equipment at Argonne National Laboratory. We collaborated first with Dr. Richard Siegel and later with Dr. Jeffrey Eastman at ANL. In the course of the studies the graduate students supported on this grant were