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# Necking contributions to $\sigma$ -phase formation in a hot-corrosion-resistant superalloy during creep exposure

J.S. Hou<sup>a,\*</sup>, L.Z. Zhou<sup>a</sup>, C. Yuan<sup>a</sup>, J.T. Guo<sup>a</sup>, W. Wang<sup>a</sup>, X.Z. Qin<sup>a</sup>, P.K. Liaw<sup>b</sup>

<sup>a</sup>Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China <sup>b</sup>Department of Materials Science and Engineering, The University of Tennessee, Knoxville, TN 37996, United States

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#### ABSTRACT

Stress has been shown to accelerate the formation of the  $\sigma$  phase in the experimental Ni-based superalloys during the high-temperature exposure. However, with the necking phenomenon occurring in strained-to-fractured specimens after the creep exposure at 1073 K/365 MPa for 1700 h, the stress decreases, while the amount of the  $\sigma$  phase increases from the fracture face to the end of the fractured gauge section. Nucleation mechanisms of the  $\sigma$  phase are discussed. The energy dynamics analysis indicates that the decrease of the  $\sigma$ -phase density by necking is mainly due to the increased matrix strain energy, while the shape, size, and composition of the  $\sigma$  phase are not affected by necking.

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#### 1. Introduction

Today's hot corrosion-resistant superalloys contain high levels of refractory elements, such as Mo and W, besides the element, Cr, in order to increase the high-temperature creep and rupture properties and obtain a favorable corrosion resistance. However, this trend can make the alloys prone to the formation of topologically close-packed (TCP) phases. Morphological changes of these Ni-based superalloys during long-term deformation at high temperatures can result in the coarsening of the  $\gamma'$ , formation of  $\gamma'$  rafts, and precipitation of the TCP phase, depending on the temperature, lattice misfit, properties of  $\gamma'/\gamma$ , and the applied stress [1-5]. With regard to the effect of stress on the TCP-phase formation, many researchers reported that stress has been shown to enhance the formation of  $\sigma$  phases [1,2], while in another case, stress has been found to delay the formation of TCP phases [3,4]. A common phenomenon called necking, occurring in the creep exposure, which influences the microstructural instability, is generally not considered. This study deals with the effect of necking on the microstructural stability of a corrosion-resistant superalloy that has been shown The experimental alloy with grain size of 1–5.5 mm is based on Ni–15Cr–11Co with Mo for solid solution strengthening and with Nb, Ti and Al for  $\gamma'$  precipitation strengthening. Phase analyses' results indicate that the weight fractions of  $\gamma', M_{23}C_6$  phase in the sample after standard heat treatment are 50.147% and 0.016%, respectively. The alloy is designed for application under 1173 K.

#### 2. Experimental

Experimental alloys were produced in an industrial-scale vacuum-induction furnace. Then the 25-kg master ingot was re-melted and cast into rods of 15 mm in diameter and 120 mm in length under vacuum. The chemical composition of the alloy in weight percent is approximately: 15.5 Cr, 10.8 Co, 5.6 W, 2.1 Mo, 3.2 Al, 4.6 Ti, 0.2 Nb, 0.4 Hf, 0.075 B, 0.073 C, and the balance Ni. The specimens used for long-term creep exposure were subjected to a standard heat treatment,

to form  $\sigma$  phases during unstressed exposures at temperatures ranging from 1073 K to 1173 K.

<sup>\*</sup> Corresponding author. Tel.: +86 24 83978469; fax: +86 24 83978045. E-mail address: jshou@imr.ac.cn (J.S. Hou).

1443 K/4 h/air cool+1323 K/4 h/air cool+1123 K/16 h/air cool. Some of the standard heat-treated bars were machined into cylindrical, threaded creep-test specimens with a gauge section of  $\phi 10~mm \times 100~mm$ . Then the samples were given constant-load tensile-creep exposures in air. The applied stress (365 MPa) is calculated with the initial cross-sectional area of the rounded bars.

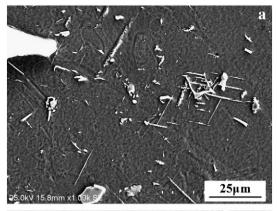
Metallography samples cut from the gauge sections of both the interrupted creep-exposed and creep-ruptured samples, which were stressed throughout the exposure, were utilized to study the microstructural stability. After the stressed exposure, the samples were unloaded and cooled to room temperature in air for microstructural characterizations. Electrochemically-etched samples were used to reveal the microstructures of σ phases by scanning electron microscopy (SEM). The measurement of the  $\gamma'$  sizes was conducted by determining the mean linear intercept of the  $\gamma'$  precipitates on the SEM images of the electrochemically-etched samples. The disks for transmission electron microscopy (TEM) observations were taken from the longitudinal gauge section, mechanically ground to ~0.1 mm in thickness, and electrochemically thinned using a twin-jet polisher. Transmission electron microscopy characterizations were used to determine the crystal structure, and the TEM supplemented with the energy-dispersive spectrometry (EDS) was used to investigate the composition of the  $\sigma$  phase.

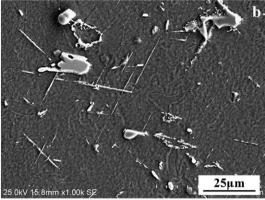
#### 3. Results and Discussion

After the stressed exposure at 1073 K/365 MPa for 1700 h, the specimen is plastically strained to fracture, and a necking phenomenon is observed. In order to analyze the necking development and growth in a cylindrical specimen subjected to a constant load, a geometrical inhomogeneity along the axis of the specimen has been modeled and considered. The shape evolution of the specimen during creep is obtained by assuming that it consists of a series of thin disks, and each disk experiences a homogeneous, uniaxial stress. This approximation is valid as long as the non-uniformity has a longer wavelength, when compared with the diameter of the specimen. Following [6], we assumed the initial cross-section has to vary along the axis of the specimen in a cosinusoidal manner. Thus, the nominal stress should be:

$$\sigma_{s} = \sigma_{s0} A_{s0} / [A_{0} - (\Delta A/2) \cos(2\pi x/l_{0})] \qquad 0 < x < l_{0}/2$$
(1)

with  $A_0$  is the nominal cross-sectional area,  $\Delta A/2$  the maximum amplitude of the perturbation,  $l_0$  the initial gauge length,  $\sigma_{s0}$  the initial applied stress,  $A_{s0}$  the initial cross-sectional area of the round specimen, and  $\sigma_{s0}A_{s0}$  the constant load. Considering the boundary conditions in the alloy, at x=0, which is the fracture face of the specimen, the cross-sectional diameter is 9.5 mm, and at x=33.2 mm $\approx l_0/3$ , which is the end of the gauge section, and the cross-sectional diameter is about 10 mm. With these values, the nominal stresses of x=0, 15, and 33 mm are calculated to be  $\sigma_s=384$ , 379, and 368 MPa, respectively. It should be noted that the nominal stress varies with the exposure time, e.g., at x=0, the nominal stress changes from 365 MPa at the onset of the creep exposure, to 384 MPa at the end of the creep exposure. Fig. 1(a), (b), and (c) illustrate the





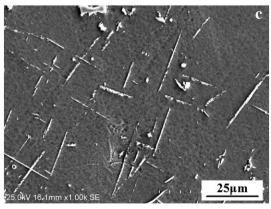


Fig. 1 – SEM photomicrographs showing  $\sigma$  phases present in the specimen with necking occurring at the position of (a) fracture face (x=0 mm;  $\sigma_s$ =384 MPa;  $f_s$ =2.3%), (b) middle (x=15 mm;  $\sigma_s$ =379 MPa;  $f_s$ =3.0%), and (c) end (x=33.2 mm;  $\sigma_s$ =368 MPa;  $f_s$ =4.8%) in the gauge section of 1073 K/365 MPa/1700 h. (x denotes the position from the fracture face to the end of the gauge section;  $\sigma_s$  is the nominal stress, and  $f_s$  is the average area fraction of the  $\sigma$  phase).

typical distributions of the deep-etched  $\sigma$  phases at x=0, 15, and 33 mm, respectively, indicating that the mean amount (an area fraction,  $f_s$ ) of the  $\sigma$  phase increases from the fracture surface ( $f_s$ =2.3%) to root (at the end of the gauge section,  $f_s$ =4.8%). Comparing the microstructures after the exposure with and without the applied stress, it is found that the exerting of stress promotes the nucleation of the  $\sigma$  phase in the alloy. At 1073 K, the stress (365 MPa) greatly decreases the time for the  $\sigma$  formation from 5000 h to 1700 h. However, in

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