Key Engineering Materials Vols. 345-346 (2007) pp. 1311-1314
online at http://www.scientific.net

A Nondestructive HKL-Planes Analysis of a Nano-Precipitate-Strengthening Alloy Study

E-Wen Huang[1, a], Yandong Wang[1, 2, b], Bjørn Clausen[3, c], Michael L. Benson[1, d], Hahn Choo[1, e], Peter K. Liaw[1, f], and Lee M. Pike[4, g]
[1]Materials Science and Engineering Department, the University of Tennessee, USA
[2]School of Materials and Metallurgy, Northeastern University, Shenyang, PRC
[3]Los Alamos Neutron Science Center, Los Alamos National Laboratory, USA
[4]Haynes International, USA
[a]ehuang1@utk.edu, [b]ydwang@mail.neu.edu.cn, [c]clausen@lanl.gov, [d]mbenson@utk.edu,
[e]hchoo@utk.edu, [f]liaw@utkux.utcc.utk.edu, [g]lpike@haynesintl.com

Keywords: In-Situ Neutron-diffraction Experiments, Nano-Precipitates, Alloy, Strengthening Mechanism.

Abstract. The HASTELLOY® C-22HS™ alloy is a corrosion-resistant nickel-based alloy in a faced-center-cubic (FCC) crystal structure. The heat-treated aging process produces the nano precipitates in the matrix and the yield strength of the aged alloy is doubled at both room temperature and 595°C. In the present study, the tensile behavior of the alloy was examined by in-situ neutron-diffraction at room temperature. The peak-width of the hkl plane from both the nano-precipitates and the matrix were determined as a function of the different applied stress. The different evolutions of the peak-widths from the matrix to the precipitates during the monotonic-tension-loading experiments clearly demonstrated that the particle-shearing mechanism. The effect of the nano-precipitate strengthening mechanism has been examined by this in-situ neutron experiments under tensile tests in this study. It reflects clearly the importance of the nondestructive in-situ neutron experiments examining the diffraction profile evolutions coupled with the mechanical measurements.

Introduction

There are research activities to study the plastic-deformation mechanisms of the nanocrystalline behaviors using the high-energy synchrotron X-ray [1, 2], high-resolution transmission-electron-microscopy [3, 4], and computer modeling [5]. Furthermore, the reliability and the performance of the newly developed nanotechnology highly rely on their mechanical properties. In this investigation, we applied the in-situ neutron-diffraction experiments to study the nano-precipitation behavior via observing the evolutions of their neutron-diffraction peak profiles at different deformation levels, because the neutron-diffraction experiments are effective for investigating the bulk properties [6]. Neutrons can penetrate deeply into the materials, and, hence, the neutron-diffractions data are able to collect from great gauge volumes [7]. The gauge volume of the present in-situ neutron experiments is 120 mm^3 and the diffraction data demonstrate the relationship between the bulk mechanical properties of the alloy and the micro behaviors of both these nanocrystalline and the matrix. The diffraction data were collected from a single specimen at different load levels. The aim of the present research is to identify the role of the nano-precipitates during the tensile loading and to illustrate how the neutron-diffraction results could be used to understand the mechanical behaviors of the nanocrystalline particles.

Testing Materials and In-situ neutron experiments

The C-22HS alloy is a commercial alloy developed by Haynes International, Inc., and the composition is Ni-21Cr-17Mo in weight percent. After a hot-rolling process, the alloy was mill annealed at 1,080°C that allows for most precipitates and alloying elements to dissolve to form a single-phase FCC alloy. The subsequent heat treatment, as 705°C/16 hours/furnace cooling (FC) and 605°C/32 hours/ air cooling (AC), produces the uniformly distributed precipitates in the FCC matrix. Moreover, after this heat treatment, the mechanical properties changes, as shown in Figure

1 [8], where the yield strength of the specimens are strengthened from the annealed to the heat-treated conditions.

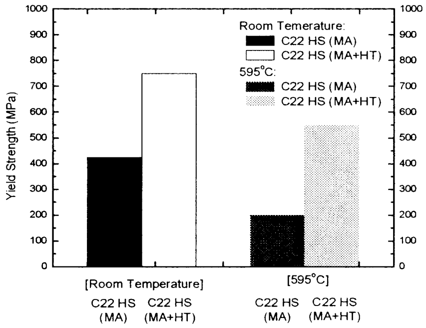

Figure 1. Comparative 0.2% yield strengths. Mill annealed (MA) = 1,080°C/Anneal/Water quench; Age-hardened (MA+HT) = MA + 705°C/16h/Furnace cool to 605°C/32h/Air cool. (After Pike.[8])

Lu et al. [9] used the transmission-electron microscopy (TEM) to study these precipitates of the alloy and found that these precipitates are in the Ni_2 (Mo, Cr) crystal structure. The volume fraction of these precipitates is 29%, and the average size is 20 nm [9]. These Ni_2 (Mo, Cr) type precipitates draw the attention because they are of a quasiperiodic structure with no forbidden symmetries [10]. However, only ex-situ experiments were conducted to observe the mechanical behaviors of these precipitates by TEM [9]. For more details, an in-situ monotonic tensile experiment coupled with neutron-diffraction experiments is necessary. In this study, the in-situ tensile experiments were performed using the Spectrometer for MAterials Research at Temperature and Stress (SMARTS) at the Los Alamos Neutron Science Center (LANSCE) [11] to observe the evolutions of the neutron diffractions from both the matrix and these precipitates. The tensile experiment was conducted at room temperature, where the strain rate is 0.001 per second. The tensile specimen was aligned 45° to the incident neutron beam with two detector banks fixed at $2\theta = \pm 90^{\circ}$. Hence, one detector collected the diffraction data along the loading direction (axial data), and the other collected the diffraction data normal to the loading direction (transverse data).

Results

The crystal structure of the alloy was measured by neutron diffractions. We used the General Structure Analysis System (GSAS) software package [12] by the Rietveld analysis [13] at LANSCE to refine the neutron-diffraction pattern. The idea of the Rietveld analysis is to refine the whole diffraction patterns based on the crystallographic structure, including the lattice parameters, volume fraction, and so on. The fitted results are shown in Figure 1. The fitting curve (line) fitted the observed data (cross) very well, which demonstrates that the structure of the alloy is indeed a single face-centered-cubic (FCC) phase matrix with the embedded Ni_2 (Mo, Cr) as the second phase. All of the matrix peaks and the precipitate peak were monitored. Among the whole diffraction patterns, there are two distinguished peaks, the 311-peak of the matrix and the 011-peak of the precipitates do not overlap to each other phase. As a result, these two peaks are better for monitoring the behaviors of the matrix and the precipitates individually. Figure 3 shows that the HKL neutron-diffraction peaks widths change as a function of the stress level during the tensile experiments. The unit of the peak-width in Figure 3 is sigma, which is the standard deviation of each individual peak and used to quantify the peak-widths. The little scatter of the precipitate peak, the solid dots, comes from the relatively smaller diffraction intensity as shown in Figure 2. The peak-widths of the matrix diffractions are depicted as hollow symbols in Figure 3. The entire matrix peaks start to broaden at the 0.2% yield strength, where the true stress is 625 MPa. Note the yield strength shown in Figure 1 is the engineering stress. The peak-width of the precipitates' 011 peak keeps the same width until 1,025 MPa.

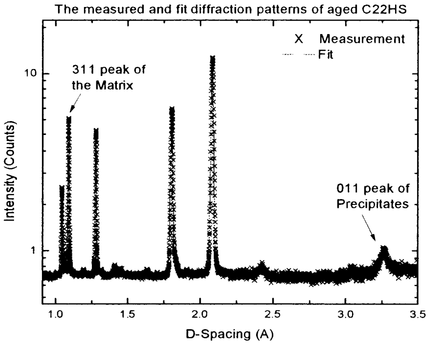

Figure 2. The Rietveld fitting of the neutron-diffraction patterns in the axial direction demonstrating that the undeformed alloy has two phases: fcc matrix and Ni_2Mo precipitates. The peak at 3.25 Angstrom is the 011 peak of the precipitates. The peaks at 2.08 Angstrom are superimposed by the 111 peak of the matrix and the 101 peak of the precipitates, the peaks at 1.80 Angstrom are superimposed by the 200 of the matrix and 121/002 peaks of the precipitates. The peak at 1.28 and 1.09 Angstrom are the 200 and 311 peaks of the matrix, respectively.

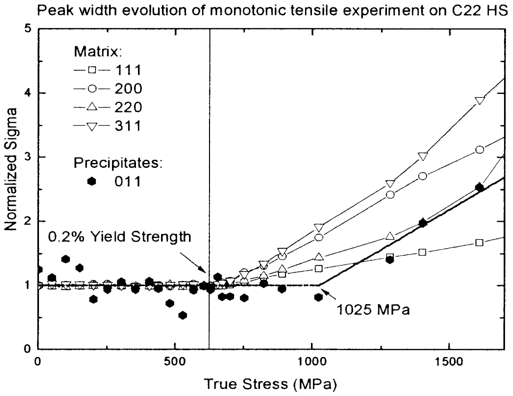

Figure 3. The peak-width evolution as a function of the true stress. The peak-widths of the matrix started to deviate around 0.2 % strain at 625MPa, where the plastic deformation began. The superlattice peak-width kept steady until 1,025 MPa, which indicates the density of the dislocation start to increase within the nano precipitates at 1,025MPa.

Discussion

The intrinsic peak-shape broadening of neutron-diffraction peaks can have several origins. Hutchings [7] and Šittner [14] reported that the evolution of the peak-width is due to the change of the number of scattering sites. Jakobsen [15] et al. reported these changes of the peak-widths came from the changes of the substructures of the grains. To sum it up, the start of the peak-width broadening reflects the beginning of the plastic deformation. More specifically, the peak-width observed in this study is mainly due to two reasons: first, the increasing of the intergranular strains due to the elastic and plastic anisotropy, and secondly the intragranular strains due to the dislocation density. In fact, because dislocations can produce severe short-range elastic strains, the peak-width could infer the dislocation density unless the dislocations are clustered into cells or boundaries [7]. Hence, we can assume that the increasing of the peak-width as shown in Figure 3 is proportional to the dislocations density in the alloy. Based on the observation of the different trends on the peak-width broadening of the matrix and the precipitates, we can conclude the present study as follows. When the true stress level smaller than 625 MPa, both the matrix and the precipitates are at the elastic deformations. At the stress level between 625 MPa and 1,025 MPa, the matrix starts to plastically deform while the precipitates is at the elastic deformation. Beyond the stress of 1,025 MPa, both of the matrix and the precipitates plastically deform. These Ni_2 (Cr, Mo) phase precipitates serve as the strengthening particles. These particles involve the interaction between mobile dislocations in the soft matrix and the hard Ni_2 (Cr, Mo) particles that act as obstacles to the motion of dislocations. Below the true stress of 1,025 MPa, the particles were not cut so that the density of dislocations kept the same within the precipitates even when the stress already exceeded 0.2% yield strength at 625 MPa. Hence we cannot observe the peak-width broadening of the precipitates before the stress level of 1,025 MPa was reached. This phenomenon is the particle-shearing mechanism, which increases the yield strength of the aged C-22HS alloy over the annealed condition, as shown in Figure 1.

Summary

The in-situ neutron-diffraction study of this monotonic tensile experiment showed a particle-shearing mechanism from the nano-size precipitates. The peak-width evolution of neutron-diffraction profiles can reflect the plastic deformation, which indicates that the increase in the dislocation density. The HKL lattice planes of both the matrix and the nano precipitates were simultaneously monitored and analyzed. This in-situ neutron study could be applied as the nondestructive technique to examine the nano-precipitation behavior.

Acknowledgement

This research is supported by the National Science Foundation (NSF), International Materials Institutes (IMI) Program (DMR-0231320). Dr. Carmen Huber is the NSF director of the IMI program. The Lujan Neutron Scattering Center at LANSCE is funded by the Department of Energy's Office of the Basic Energy Science. The Los Alamos National Laboratory is operated by the Los Alamos National Security LLC under the DOE Contract DE-AC52-06NA25396. We appreciate Dr. Dwaine L. Klarstrom's great support on the materials from Haynes International, Inc. We are thankful for the discussions with Dr. Donald W. Brown and Dr. Tarik Saleh at LANSCE and we are grateful for Mr. Soo-Yoel Lee's kind help.

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