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Internal friction studies of carbon and its redistribution kinetics in Inconel 600 and 690 alloys under dynamic strain aging conditions

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Abstract

Carbon Snoek-like relaxation peak with a height of about 10^{-4} was found in annealed nickel-based Inconel 600 alloy at 620–670 K while no carbon-induced peaks were observed in Inconel 690 alloy. The activation enthalpy and the pre-exponential factor of the relaxation time are 1.68 ± 0.09 eV and $10^{-14.14 \pm 0.72}$ s, respectively. The height of the observed peak increases dramatically, up to about 10^{-3} , after tensile deformation of the Inconel 600 alloy at 423 K at the strain rate of 10^{-5} s $^{-1}$, when dynamic strain aging (DSA) behavior exhibits well developed serrations on the tensile stress–strain curve. Annealing *in situ* at the carbon peak temperature results in decay of the peak amplitude, which consists of two exponential stages with characteristic times of 1.6 and 21.9 ks, respectively. The first characteristic time is in good agreement with the mean period of DSA serrations. The second characteristic time probably presents the re-growth of carbides partly dissolved during prior deformation in DSA conditions.

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1. Introduction

Carbon plays an important role in the engineering properties of Inconel 600 and 690 alloys. Strength and creep resistance of these alloys in the environmental conditions of nuclear reactors depend on the amount, state and diffusion mobility of carbon dissolved in the alloy. Because of the high Cr content (about 16 and 30 wt.% for Inconel 600 and 690, respectively) in the industrial nickel-based alloys, major part of carbon resides in carbides and only a small fraction exists as interstitial solution. Because of the interaction of carbon with mobile dislocations, even a minor content in the solid solution results in dynamic strain aging (DSA) of Inconel alloys at temperatures above 423 K [1,2], which can markedly affect the environmentally assisted cracking susceptibility of these alloys in high temperature water conditions of nuclear reactors.

Information about atomic state of interstitial carbon and its elemental diffusion jumps in multicomponent nickel-based alloys is the prerequisite for a better understanding and modeling of the behavior of Inconel alloys and their degradation under real

reactor conditions. The necessary information on carbon behavior can be obtained by using mechanical spectroscopy methods. Internal friction measurements of the carbon-induced anelasticity have been performed for many austenitic stainless steels, nickel, substitutional nickel-based alloys and for concentrated Ni–Co alloys; see, for instance, a recent brief review [3].

In spite of the great practical importance, no systematic internal friction studies of carbon behavior in Inconel 600 and 690 alloys have been performed for the present. The aim of this study is to verify the presence of the carbon Snoek-like peak in these nickel-based alloys and to obtain its relaxation parameters. Additional attention is paid to the effects of plastic deformation under DSA conditions on the state of carbon in Inconel 600 alloy by studying the evolution of Snoek-like peak of carbon.

2. Experimental

Two industrial grades of nickel-based Inconel 600 and 690 alloys were used in the study. Chemical compositions of the alloys are shown in Table 1.

Alloys were supplied as plates in an annealed condition with thickness of about 14 mm for Inconel 600 and 5 mm for Inconel 690. The average grain size was about 100 μ m for Inconel 600 alloy and about 30 μ m for Inconel 690 alloy. The materials were

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Table 1
Chemical compositions (in wt.%) of Inconel alloys

Alloy	C	Cr	Fe	Ti	Nb
Inconel 600	0.04	16.8	8.1	0.16	0.01
Inconel 690	0.01	27.7	8.4	0.14	0.01

cut using water-jet cutting and spark-erosion techniques to form flat specimens for mechanical tensile tests with a cross-section of 1.9 mm × 6 mm and a gauge length of 32 mm. A 25 kN MTS 858 testing machine equipped with a split furnace was used for tests at elevated temperatures. Tensile test specimens were strained to fracture (strain about 50% [2]) at test temperature and strain rate of 10^{-5} s^{-1} . Cracking localized close to the end of the gauge length provided enough material with uniform elongation for internal friction measurements. Cooling of the samples from test to room temperature did not exceed 1.5 min. Specimens for internal friction measurements with typical sizes of about 0.5 mm × 2 mm × 35 mm were cut from the gauge part of the tested specimens by abrasive disc-saw. Some other specimens were cut from as-supplied annealed materials. All specimens were polished with 1200 grit emery paper.

Temperature dependencies of internal friction Q^{-1} and natural frequency of the pendulum were measured using the method of free decay of resonance oscillation by an inverted torsion pendulum in the temperature range from 300 to 900 K with strain amplitudes less than 10^{-5} . The pendulum frequency was changing in the range from 0.3 to 3 Hz. The heating rate was 2 K/min.

3. Results and discussion

Temperature dependencies of internal friction and squared natural frequency for as-supplied Inconel 600 and 690 alloys are shown in Fig. 1. A distinct peak in annealed Inconel 600 alloy is present at about 660 K, while no peaks were observed either in annealed or in deformed Inconel 690 alloy. The peak in Inconel 600 alloy arises in the temperature range where carbon-induced Snoek-like peaks have been observed in austenitic stainless

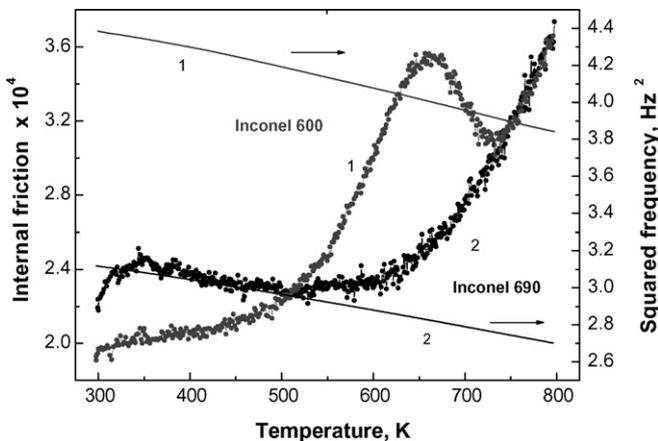


Fig. 1. Temperature dependencies of internal friction and squared natural frequency for Inconel 600 (1) and Inconel 690 (2) alloys in as-supplied, annealed state.

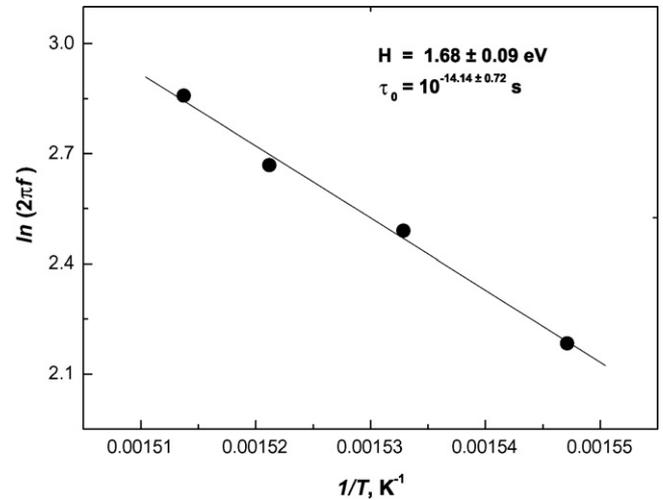


Fig. 2. Arrhenius plot of the activation analysis for carbon peak in annealed Inconel 600 alloy.

steels [3] and in nickel [4], and therefore they are assumed to be of the same origin.

The carbon peak in Inconel 600 alloy was recognized to be based on a relaxation process by using standard activation analysis with shifts of the natural frequency of the pendulum by inertia members. The Arrhenius plot for this analysis is shown in Fig. 2. The activation enthalpy and pre-exponential factor of the relaxation time are $1.68 \pm 0.09 \text{ eV}$ and $10^{-14.14 \pm 0.72} \text{ s}$, respectively. The obtained activation enthalpy is close to the activation energy for carbon diffusion in nickel at higher temperatures, which was found to be 1.54 eV [5], but is slightly different from the activation energy for carbon diffusion in Inconel 600 alloy obtained from DSC analysis of chromium carbide precipitation at 873 K, 1.43 eV [6].

Prior plastic deformation of Inconel 600 alloy in the temperature range above 423 K, where DSA starts in the form of jerky flow, results in a significant increase of the carbon peak height and its maximum shifts to lower temperatures, as shown in Fig. 3. No peaks were observed in this temperature range in

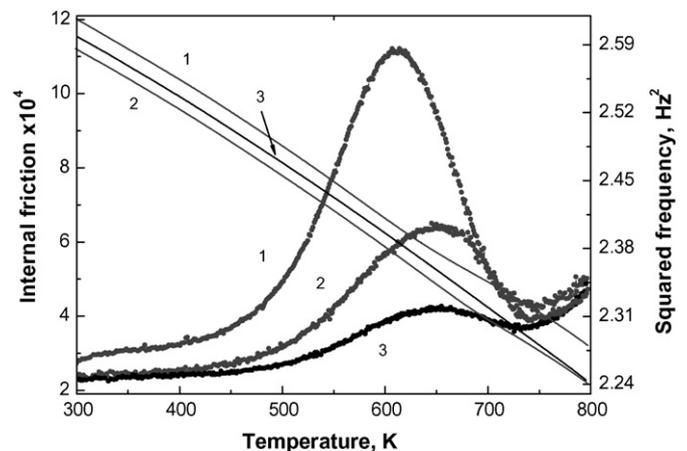


Fig. 3. Effect of prior DSA deformation at 573 K and strain rate of 10^{-5} s^{-1} (1) and subsequent annealing at 620 K for 7 h (2) on the carbon peak in Inconel 600 alloy; curves (3) correspond to the peak in as-supplied, annealed state.

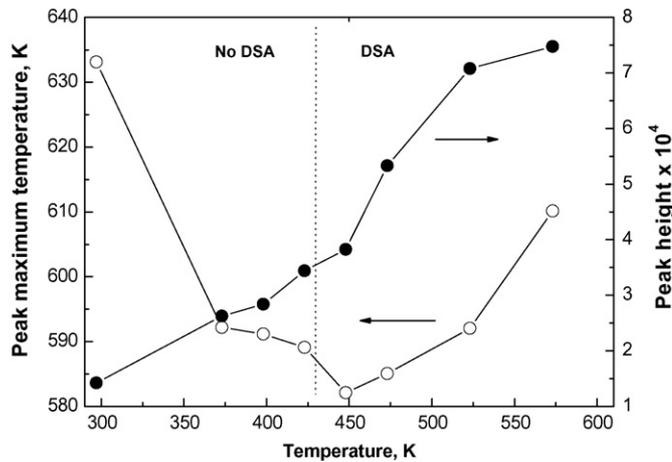


Fig. 4. Dependencies of the carbon peak temperature (open circle) and height (filled circle) on temperature of prior tensile testing with strain rate of 10^{-5} s^{-1} . The dotted line depicts the temperature of DSA appearance.

Inconel 690 alloy neither for as-supplied state nor for deformed condition.

The maximum peak temperature and its height for a series of prior tensile testing temperatures are shown in Fig. 4. The temperature position of the peak shifts down for 50 K in comparison to its position for the annealed state of the material, and the height becomes three times higher when the prior deformation temperature approaches the DSA regime. The peak height saturates and its temperature position returns to higher temperatures at tensile testing temperatures above 523 K, probably because of partial recovery of the observed effect during cooling from higher tensile test temperatures. The position and height of the peak return to their original values of as-supplied state of Inconel 600 alloy during *in situ* annealing, as shown in Fig. 5. The kinetics of peak height recovery fits well with the sum of two exponents with characteristic times of 1.6 and 21.9 ks. The first characteristic time corresponds well to the period of the jerky flow of the Inconel 600 alloy observed under DSA regime of deformation [2].

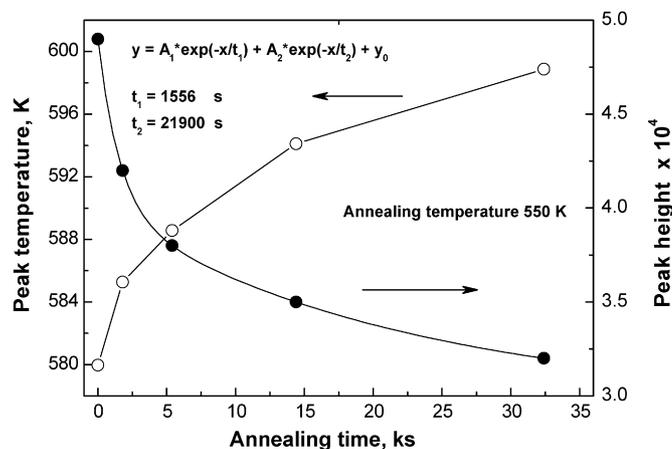


Fig. 5. Temperature position and height of the carbon peak in Inconel 600 alloy deformed at 473 K and strain rate of 10^{-4} s^{-1} as a function of time of annealing at 550 K. Decay of the peak height fits well with the sum of two exponents with characteristic times of 1.67 and 21.8 ks.

Similar effects of prior deformation on the carbon peak have been observed in austenitic stainless steels [7]. The increase of the carbon peak by deformation in austenitic stainless steels originates from the specific state of carbon in the FCC lattice of austenite enhanced by strain fields of dislocations. At the same time, it was noticed that the binding energy of interstitial carbon to a dislocation in this case is much lower than in the case of BCC iron, where deformation results in the well-known carbon-dislocation Snoek–Köster peak. The interpretation of the deformation effects on the carbon peak in multicomponent iron- and nickel-based FCC alloys proposed by Blanter et al. [7] does not explain the transitional character of the observed phenomenon shown, for instance, in Fig. 5.

A significant increase of the peak height above the temperature of DSA occurrence observed in the present study evidences that the discussed effect can be caused by deformation-induced microscopic re-distribution of carbon. Localization of plastic deformation, which is a feature of DSA, leads to accumulation of carbon in the regions of intensive plastic flow. In other words, some microscopic areas become enriched with carbon atoms while depletion takes place elsewhere. One can also assume that, in Inconel 600 alloy, carbon can form two different states. The first one can be similar to that in unalloyed nickel, namely, carbon–carbon pairs, while the second state is a single interstitial in an octahedral site next to a solute atom of Cr or other carbide-forming elements such as Ti or Nb. The partitioning to these states depends on the local concentration of carbon. In enriched areas the first state prevails, whereas the second state is preferred for the homogeneous and low carbon concentration in the case of annealed alloy or within depleted zones. Finally, if the carbon–carbon pair diffusion (re-orientation) is faster than that of the single carbon interstitial and if the pair induces higher relaxation strength than that of two single carbon states, deformation in DSA conditions can result in a shift of the carbon peak to a lower temperature with a remarkable increase in height because of the change of the origin of relaxation. During annealing at temperatures where carbon becomes mobile, the enriched areas disappear and the initial (before deformation) low and homogeneous carbon concentration is established. This can explain the decay of the carbon peak observed. The first characteristic time of decay, 1.6 ks, corresponds, at temperatures of about 550 K, to the diffusion distances of carbon of about $0.3 \mu\text{m}$. The second characteristic time may reflect escape of carbon from the solid solution to carbide particles, which are present mainly at grain boundaries.

An alternative explanation for the deformation effect on the carbon peak may be the anelastic response of carbon-vacancy pair [8]. Actually excessive vacancies may be generated by plastic deformation under DSA and they can affect the carbon peak.

Two times higher chromium content in Inconel 690 alloy than in Inconel 600 alloy is probably the origin of the absence of the carbon peak. Chromium reduces markedly the solubility of carbon in nickel-based alloys [9] and increases the number of octahedral interstitial sites coordinated with six Cr atoms. Because of their high affinity to chromium, carbon atoms may be strongly bound to Cr atoms and they do not induce any measurable anelastic response in Inconel 690 alloy.

4. Conclusions

An internal friction peak induced by carbon is observed in Inconel 600 alloy at about 650 K for frequencies around 1 Hz. The peak, however, is absent in Inconel 690 alloy, which contains two times higher amount of chromium than Inconel 600 alloy.

The observed carbon peak has a relaxation origin with an activation enthalpy of 1.68 ± 0.09 eV and a pre-exponential factor of the relaxation time of $10^{-14.14 \pm 0.72}$ s.

Prior plastic deformation increases markedly the height of the carbon peak and shifts its position to lower temperatures. The effect depends on the deformation temperature and becomes marked at temperatures where DSA occurs in Inconel 600 alloy.

Plastic deformation in the DSA regime results in an inhomogeneous distribution of carbon in the solid solution. Carbon–carbon pairs forming in carbon-enriched zones explain probably a major part of the observed low temperature internal friction peak. Single carbon interstitials or their pairs with Cr, Nb or Ti atoms are responsible for the peak in annealed Inconel 600 alloy.

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