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Application of the internal friction method to studying microstructural effects in fusion materials

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Abstract

Two candidate materials for the blanket components of ITER were studied by the internal friction method. For low-activation martensitic F82H steel measurements of anelasticity were carried out in the temperature range of 80–500 K after preliminary electrolytical hydrogen charging. A broad multicomponent internal friction peak was detected in hydrogen-charged F82H steel in the temperature range of 100–450 K at a frequency of 1 Hz. The effect of the amount and the type of strengthening particles on the internal friction were clarified for CuAl25, CuAl60 and CuAl25 + TiC GlidCop™ dispersion strengthened copper alloys in the temperature range of 300–1000 K. The effect of neutron irradiation on internal friction of CuAl25 alloy is also discussed. © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

Operation conditions of ITER impose very strict requirements to the physical and mechanical properties of the engineering materials for the first wall and the divertor components. The demanded combination of strength, fracture toughness and thermal conductivity can be achieved by producing special microstructural states of alloys. Special attention should be paid to the stability of the formed microstructures under the ITER operation conditions – at elevated temperatures under neutron irradiation.

Internal friction method is based on the study of anelastic response caused usually by interstitial behavior [1]. The method is particularly useful in studying point defect behavior and, especially, their interaction with dislocations. Thus, the effects of defects generated by irradiation can be successfully studied with the internal friction technique.

Neutron irradiation as well as hydrogen charging are known to enhance degradation of mechanical properties of martensitic steels [2]. Because of penetration of hydrogen into the first wall material from plasma or coolant, as well as accumulation as a result of transmutation reactions, hydrogen embrittlement may occur or hydrogen atoms may interact with carbon leading to hydrogen attack, i.e., to the development of gaseous porosity. Internal friction studies of hydrogen effects have revealed a multicomponent peak in hydrogen-charged martensitic F82H steel [3]. The structure of the peak indicates a complex behavior of hydrogen atoms in the martensite lattice and its possible interaction with dislocations.

The size and distribution of alumina particles determine to a great extent the mechanical properties of GlidCop™ copper alloys. Recent experimental results indicate that these alloys suffer loss of ductility, strain hardening capacity and toughness at elevated temperatures [4,5]. The internal friction studies of CuAl25 alloy have revealed a broad two component peak in the temperature range of 300–850 K [6]. The low temperature part of the internal friction peaks is expected to be a grain boundary effect possibly due to grain boundary sliding [1] and the high temperature part is expected to arise due to anelastic response of the particle matrix interfaces. Neutron irradiation has been shown to suppress the low temperature part of the internal friction peaks when compared to unirradiated condition [6].

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The present work is devoted to the internal friction study of the fine structure of the earlier detected hydrogen peak in low-activated martensitic F82H steel and to the comparative study of the internal friction of different type of GlidCop™ copper alloys.

2. Experimental

Temperature dependencies of internal friction Q^{-1} and frequency f were obtained with inverted torsion pendulum in the temperature range of 80–500 K for the martensitic F82H steel and 300–1000 K for the GlidCop™ copper alloys. The frequency of natural oscillations varied in the range of 0.3–3 Hz by varying the torsion stiffness of the pendulum (cross-section of the samples). Heating rate was kept constant 0.5 K/min for the hydrogen-charged F82H steel and 2 K/min for the copper alloys. Torsion strain amplitude did not exceed 10^{-5}.

Martensitic F82H steel containing 8 wt% Cr, 2 wt% W and 0.1 wt% C was supplied by NNK-JAERI (Japan). The billet underwent the complete cycle of thermal treatment including multipass hot rolling at 1250°C, normalization at 1040°C for 37 min, quenching and tempering at 750°C for 1 h, and furnace cooling.

Internal friction samples of F82H steel with typical dimensions of 0.5–1 × 1–2 × 40 mm^3 were cut from an industrial billet in the as-supplied condition and after 10, 20 and 27% cold-rolling. The samples were electrolytically hydrogen charged in 1N $H_2SO_4 + 5$ mg/l $NaAsO_2$ solution with the charging current density of 20 mA/cm^2 at room temperature. The duration of hydrogen charging was 10 and 13 h, and the hydrogen content was determined using Leybold–Heraeus H_2A 2002 analyzer and was found to be 5 and 11 ppm for the as-supplied and cold-worked (27%) conditions, respectively.

Three types of GlidCop™ copper alloys (produced by OMG Americans), GlidCop®Al-25 IG0, GlidCop®Al-60 and GlidCop®Al-25 + TiC, were studied. The first two alloys contained 0.46 and 1.1 wt% of alumina particles with the average size of 10 nm (0.1 μm at grain boundaries) and 100 nm (1 μm at grain boundaries), respectively. The last alloy contained TiC particles in addition to 0.46 wt% of Al_2O_3. In all three alloys, the strengthening particles were unevenly distributed in the bulk of the grains. The grain structure was strongly elongated and the average grain size reached about 20 μm in the direction of rolling and was about 1 μm in diameter in the transverse direction.

Internal friction samples of GlidCop™ copper alloys with typical dimensions of 0.5–1 × 1–2 × 40 mm^3 were cut with a diamond saw from industrial billets. The finishing was performed with 800 grit emery paper.

3. Results and discussion

Temperature dependencies of internal friction Q^{-1} for martensitic F82H steel are given in Fig. 1. A spread multicomponent peak in the temperature range of 100–450 K was observed after cold-rolling and subsequent hydrogen charging. The height of the peak increased with increasing amount of preliminary cold-work.

Analyses of the internal friction spectra for the cold-worked and hydrogen charged steel indicate that the low-temperature part of the peak (below 250 K) consists of several components. Fig. 2 shows how the internal friction spectrum can be described with three components. The high-temperature part of the spectrum is, apparently, caused by the hydrogen redistribution and desorption at elevated temperatures [3].

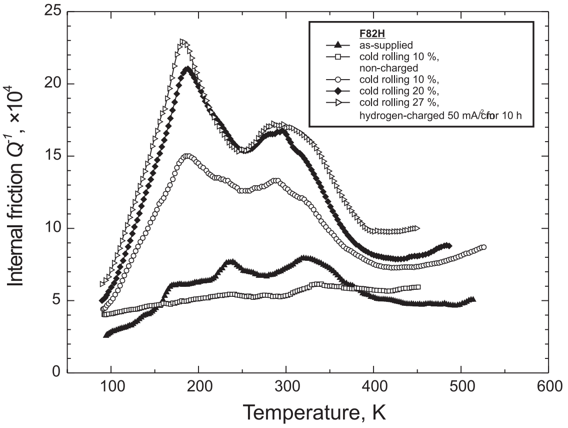

Fig. 1. Temperature dependencies of internal friction Q^{-1} for as-supplied and 10, 20 and 27% cold-rolled F82H steel with and without hydrogen charging.

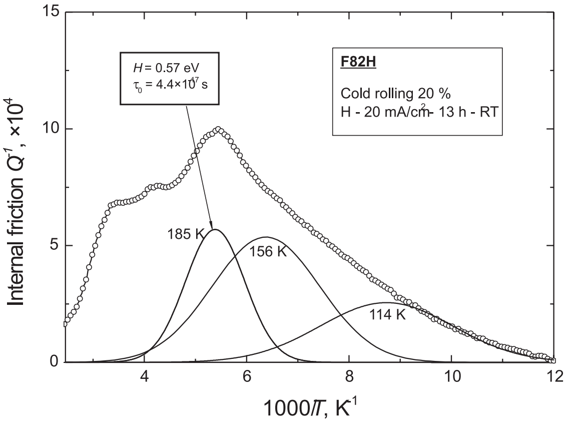

Fig. 2. An example of deconvolution of the peak observed in 20% cold-rolled hydrogen charged F82H steel to components.

Assuming Arrhenius dependence of the relaxation times, the apparent activation parameters were determined for the 185 K component (see Fig. 3). The activation enthalpy was determined to be $H = 0.57$ eV, and the pre-exponential factor of the relaxation time to be $\tau_0 = 4.4 \times 10^{-17}$ s. Activation analysis of the other components is hampered since the temperature positions of the components can be determined only roughly. Assuming for the components at approximately 150 and 110 K, however, hydrogen Snoek-like relaxation and using $\tau_0 \propto 10^{-13}$ s as the pre-exponential factor of the relaxation time, the value of $H \approx 0.37$ eV can be obtained for the apparent activation enthalpy. It is, also, assumed that in the vicinity of the hydrogen peak, i.e., at $T_p \approx 150$ K, the condition $\omega\tau_0 \exp(H/k_BT) \approx 1$ is valid [1], where k_B is Boltzmann's constant.

The obtained values of the activation parameters for the 185 K component and the strong dependence of its height on the amount of preliminary cold-work allow to assume that this component is the Snoek–Köster relaxation peak [1], i.e., it is caused by the anelastic response of dislocations interacting with mobile hydrogen atoms. According to the Schoeck model [8], the activation enthalpy of the Snoek–Köster relaxation is $H_{SK} = H_b + H_d$, where H_b is the binding energy of a hydrogen atom to a dislocation, and H_d is the apparent enthalpy of hydrogen diffusion. The obtained value $H \approx 0.37$ eV of the activation enthalpy of the Snoek-like component at approximately 150 K can be regarded as an estimated value for the H_d. Thus, for the binding energy H_b in F82H steel the value of $H_b \approx 0.21$ eV can be obtained, which is in good agreement with other data [9].

The binding energy H_b is one of the main parameters determining the development of hydrogen-enhanced localized plasticity as an initial stage of hydrogen embrittlement [10] through the formation of the shielding hydrogen atmospheres on dislocations. For a given average hydrogen concentration in solid solution, c, the temperature at which the hydrogen Cottrell atmosphere becomes dense, i.e., attains the value of $c_d \approx 1$, can be estimated as $T_c \approx H_b/k_B \ln c$ (concentration c in H/Me units). For the concentration of about 10 ppm (5.6×10^{-4} in H/Me units), temperature T_c equals to about 300 K. Thus, above room temperature, the hydrogen atmospheres are diluted in F82H martensitic steel and should not have any shielding effects on dislocation interactions and hydrogen embrittlement by the hydrogen enhanced localized plasticity (HELP) mechanism.

Temperature dependencies of internal friction Q^{-1} for GlidCop™ CuAl25 IG0, CuAl60 and CuAl25 + TiC copper alloys are shown in Fig. 4. The temperature dependence of internal friction for neutron irradiated CuAl25 IG0 is given for comparison [6]. The qualitative behavior of internal friction spectrum does not change as compared with CuAl25 IG0 alloy, neither with increasing number of strengthening alumina particles (CuAl60), nor with introduced TiC particles. In all cases, a wide two-component peak is observed.

It is suggested that the low-temperature part of the spectrum is caused by grain boundary effects, e.g., grain boundary sliding or grain boundary dislocation movement, while the high-temperature part is controlled by anelastic response of the matrix-strengthening particle interfaces [6,7]. Increasing amount of the strengthening particles suppresses the high-temperature part of the internal friction spectrum in the temperature range of 600–850 K. The highest effect was observed on the alloy with TiC particles. On the other hand, introduction of TiC particles seems to enhance the low-temperature peak (e.g., grain boundary sliding), in the temperature

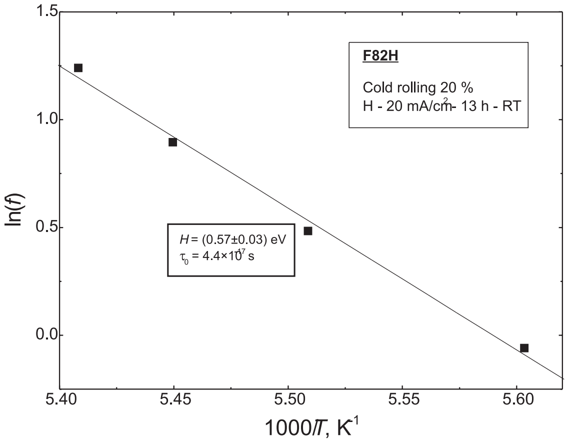

Fig. 3. Arrhenius plot for the 185 K component of the experimental internal friction spectrum of 20% cold-rolled hydrogen charged F82H steel.

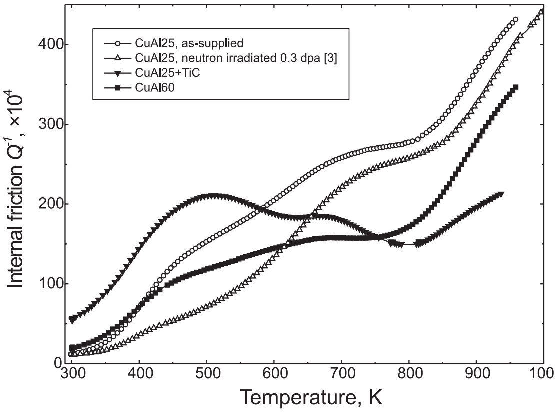

Fig. 4. Temperature dependencies of internal friction Q^{-1} for GlidCop™ CuAl25, CuAl60 and CuAl25 + TiC copper alloys.

range of 300–600 K, while increasing amount of alumina particles or neutron irradiation seems to suppress the low-temperature peak.

Alumina particles at grain boundaries are expected to be more coarse and their density to be higher in CuAl60 as compared to CuAl25 alloy. This is expected to result in more effective suppression of grain boundary effects like sliding or grain boundary dislocation movement in the former alloy, which is also observed by the suppressed low temperature internal friction peak. Recent experimental results indicate that the fracture toughness of CuAl60 is significantly lower whereas that of CuAl25 + TiC is slightly higher than the fracture toughness of CuAl25 alloy. The fracture mode in these alloys also changes from ductile dimple to grain boundary type of fracture at temperatures above 200°C [11].

The fact that neutron irradiation suppresses the low temperature internal friction peak below 600 K (see Fig. 4) and results, at the same time, in a drastic reduction of fracture toughness [5,6,12] allows to assume that there may exist a connection between grain boundary effects observed by internal friction and fracture toughness behavior. This is also consistent with the introduction of TiC particles, which enhances the low temperature internal friction peak and results in slightly better fracture toughness [11]. Indeed, if grain boundary effects, like grain boundary sliding, is an additional mechanism of plastic deformation in GlidCop™ type of copper alloys, its operation may offer a mechanism for tailoring alloys with improved fracture toughness properties under the ITER exploitation conditions.

4. Conclusions

The performed internal friction measurements on low-activation F82H steel and GlidCop™ copper alloys have shown the effectiveness of the method in determining the parameters of interaction of crystal lattice defects and fine microstructural changes in these materials.

The binding energy of hydrogen to dislocations in F82H steel has the value of 0.21 eV, which is the key parameter in the modeling of hydrogen embrittlement of martensitic steels under the ITER operation conditions.

The critical temperature of the formation of dense hydrogen Cottrell atmospheres on dislocations was estimated as 300 K, which makes hydrogen enhanced localized plasticity to be a very unprobable mechanism of hydrogen degradation at ITER operation temperatures.

The amount of anelastic response of GlidCop™ copper alloys depends on the volume density and type of strengthening particles. Increase of the size of alumina particles and their density suppresses grain boundary sliding, while introduction of TiC particles enhances significantly grain boundary sliding. It is suggested that the ability to grain boundary sliding and the fracture toughness behavior have a correlation. The detailed mechanism requires, however, further studies.

Acknowledgements

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