



ELSEVIER

Journal of Nuclear Materials 275 (1999) 47–55

Journal of  
nuclear  
materials

www.elsevier.nl/locate/jnucmat

# Internal friction study of hydrogen behaviour in low activated martensitic F82H steel

Yu. Jagodzinski<sup>a</sup>, A. Tarasenko<sup>a</sup>, S. Smuk<sup>b,\*</sup>, S. Tähtinen<sup>c</sup>, H. Hänninen<sup>b</sup>

<sup>a</sup> Institute for Metal Physics, National Academy of Sciences, Vernadsky blvd. 36, 252142, Kiev, Ukraine

<sup>b</sup> VTT Manufacturing Technology, P.O. Box 1704, FIN-02044 VTT, Finland

<sup>c</sup> Laboratory of Engineering Materials, Helsinki University of Technology, P.O. Box 4200, FIN-02015 TKK, Finland

Received 17 July 1998; accepted 28 March 1999

## Abstract

Hydrogen behaviour in martensitic F82H steel was studied by internal friction. A broad internal friction peak was found in the temperature range of 100–450 K. It was shown that the peak consists of one relaxation component and two components corresponding to the transient processes of the hydrogen redistribution and outgassing. The relaxation component of the peak is caused by the Snoek-like process of the transition of hydrogen atoms between the tetrahedral interstitial sites coordinated with one and/or two chromium atom nearest neighbours. The binding energy of hydrogen to the lattice traps is 33 kJ/mol. Cold working was shown to increase the hydrogen absorption capability of the material significantly by production of additional hydrogen traps. © 1999 Elsevier Science B.V. All rights reserved.

PACS: 61.72.Hh; 61.72.Ss; 62.40.+i

## 1. Introduction

Ferritic–martensitic steels with 9–12 wt% Cr have been intensively studied during the last decade as candidates for the engineering materials of the first wall and blanket of the fusion reactor. Favourable mechanical properties as compared to austenitic stainless steels, radiation and corrosion resistance, structural stability, thermal conductivity, as well as absence of nickel allowing to avoid neutron-induced radioactivity, make these steels an excellent choice for applications in the nuclear industry.

Hydrogen is one of the most strongly affecting factors for the first-wall material. Hydrogen can penetrate into the metal from plasma and coolant, or be produced as a result of transmutation reactions [1]. There exists a danger of hydrogen attack at elevated temperatures, which may lead to methane formation in voids, since the steels contain up to 0.1 wt% C. A synergistic effect of

irradiation and hydrogen seems to be possible, as well. Indeed, the binding energy of a hydrogen atom with a vacancy in BCC iron is about 58 kJ/mol [2]. The value of the binding energy should be approximately the same in these steels. Then, the hydrogen–vacancy complexes are rather stable, at least at moderate temperatures. This should result in a significantly higher amount of vacancies, as compared to the thermodynamical equilibrium in the absence of hydrogen, which may, in turn, enhance and accelerate vacancy-driven structural changes in the metal, e.g., low-temperature creep.

Cathodic charging of 9–12 wt% Cr steels in typical charging conditions [3], results in 5–10 wt ppm of total hydrogen content. The value of 6 wt ppm of total hydrogen content in HT-9 steel leads to a decrease of reduction of area (RA) from 60% to 15% [4]. The decrease of RA is accompanied with a transition to intergranular fracture. In martensitic 9Cr–2W steels, the value of elongation to fracture decreases from 19% to 5% after cathodic charging [5]. Hydrogen embrittlement is believed to be caused by the reduction of cohesion between carbide particles and the matrix, as well as by the accumulation of hydrogen at interfaces in the martensitic steels.

\* Corresponding author. Tel.: +358-9 451 3934; fax: +358-9 451 3537; e-mail: sergiy.smuk@hut.fi

Hydrogen, along with irradiation, high temperature and coolant environment, affects the first wall materials. Understanding of the degradation mechanisms in low-activated steels (LAS) demands for the study of hydrogen interaction with Fe–Cr alloys, i.e., distribution of hydrogen in BCT lattice of martensite and hydrogen interaction with crystal lattice defects, such as dislocations and carbide–matrix interfaces.

Deformation by tension and cold rolling affects the hydrogen permeation in chromium-containing steels and results in an increasing number of reversible traps [6]. Dislocations are believed to be such traps. There is no detailed information on the interaction of hydrogen with the crystal lattice defects in LAS, so far. It may only be assumed that the values of the energy of hydrogen-defect interactions are close to those in pure iron. In pure iron and ferritic steels, the binding energy of a hydrogen atom with dislocations and grain boundaries was evaluated to be 20–30 and 59 kJ/mol, respectively [7].

Interactions of hydrogen with dislocations was studied in Fe–6.2 at.% Cr and Fe–27.9 at.% Cr alloys, as well as SUS 410 and SUS 430 steels by internal friction technique [8,9]. Relaxation peaks were detected in electrochemically charged Fe–Cr alloys at the frequency of 500 Hz and the temperatures of 200–250 K. These peaks were recognised as Snoek–Köster peaks. The enthalpies of activation for these peaks were calculated by the frequency shifts and equalled to 71 and 73 kJ/mol for chromium content of 6.2 and 27.9 at.%, respectively. The enthalpy of hydrogen interaction with dislocations was calculated to be 32 and 45 kJ/mol [9], taking into account the values of 39 and 28 kJ/mol of the enthalpy of hydrogen diffusion in Fe–6.2 at.% Cr and Fe–27.9 at.% Cr alloys, respectively.

The results obtained by Asano et al. [8,9] demonstrated that the internal friction technique is an effective tool in studying hydrogen interaction with dislocations in ferritic Fe–Cr alloys. The present study was aimed at studying low-frequency internal friction in hydrogen-charged low-activated martensitic F82H (9Cr–2W) steel.

## 2. Experimental methods

In the present study, samples obtained from NNK–JAERI (Japan) were investigated. Chemical composition of the material is given in Table 1.

Samples were cut from an industrial ingot, which had undergone the complete cycle of metallurgical and thermal treatments. Slabs were hot rolled at 1250°C in several steps of 25% of strain each with intermediate annealing at 760°C in vacuum for 2 h. Final treatment included normalisation at 1040°C for 37 min followed by fast cooling and tempering at 750°C for 1 h followed by furnace cooling.

Temperature dependencies of internal friction (IF)  $Q^{-1}$  and shear modulus of the steel were measured by torsion pendulum in the temperature range from the liquid nitrogen temperature to 500 K at the frequency of natural oscillation around 1 Hz. The torsion strain did not exceed  $10^{-5}$ . The constant heating rate varied from 0.5 to 3 K/min. Longitudinal tension load on the sample in the pendulum was about 4 N.

Internal friction in F82H steel was studied in the as-supplied condition and after cold rolling by 10%, 21% and 27%. After treatments, samples were cut by a diamond saw, polished by emery paper and, finally, electropolished in 860 ml  $H_3PO_4$  + 100 g  $CrO_3$  solution. The final shape of the samples was a bar with the dimensions of  $0.3\text{--}1.0 \times 2 \times 45 \text{ mm}^3$ . The thickness of the samples was varied to provide different torsion stiffness and, thus, different frequency of oscillation.

Hydrogen charging was performed in 1N  $H_2SO_4$  + 5 mg/l  $NaAsO_2$  solution with a current density of 50 mA/cm<sup>2</sup> in a thermostat cell with a platinum anode. The values of hydrogen content after different charging times and for different values of cold deformation are given in Table 2. The measurements were performed with a Leybold–Heraeus RH<sub>2</sub>–2000A analyzer.

After hydrogen charging had been completed, IF samples were stored in liquid nitrogen before measurement. The time of the installation of the sample into the

Table 1  
Chemical composition of the studied material (wt%)

C	Si	Mn	P	S	Cu	Ni	Cr	Mo	V	Nb	B	N	Sol. Al	Co	Ti	Ta	W
0.09	0.11	0.16	0.002	0.002	0.01	0.02	7.64	0.003	0.16	0.0001	0.0002	0.006	0.003	0.005	0.01	0.02	1.94–1.97
							–7.71					–0.008					

Table 2  
Hydrogen contents of F82H steel after hydrogen charging in 1N  $H_2SO_4$  + 5 mg/l  $NaAsO_2$  solution with a current density of 50 mA/cm<sup>2</sup> after different charging times and degrees of deformation

Condition	As-supplied					Cold-rolled, 10%		Cold-rolled, 21%
Charging time (h)	1	3	5	12	24	10		10
Hydrogen content (wt ppm)	4.61	5.5	4.94	5.05	5.13	9.7		11

relaxometer and cooling it down to approximately 80 K did not exceed 4 min.

### 3. Results

As shown in Fig. 1, F82H steel did not reveal any clear IF peak in the temperature range of 80–500 K in the as-supplied condition. Only a weak broad hump was detected at the temperatures of 250–400 K. This hump disappeared after the second measurement of the same sample (second run). After hydrogen charging, F82H steel revealed a wide IF peak in the temperature range of 100–400 K. Three components, located at the temperatures of approximately 185, 260 and 340 K, can be distinguished. For convenience, the components will be designated as P1, P2 and P3, respectively.

Fig. 1(b) shows that the shear modulus in hydrogen charged F82H steel revealed a drop in the temperature range of 170–350 K, followed by a modulus recovery to its normal behaviour in the hydrogen-free material. Such behaviour is typical for hydrogenated F82H steel and was observed in all the studied cases.

The conditions of hydrogen-charging used in the present study result in a damaged surface layer of the steel. In turn, this leads to a decrease of the torsion stiffness of the sample, which can be considered as the reason for the decrease of the apparent, or effective, shear modulus. Such a decrease of the apparent modulus would be irreversible and independent of temperature.

Therefore, the observed drop of the frequency squared in the vicinity of the peak must be caused by anelastic behaviour of the F82H steel lattice containing hydrogen.

Fig. 2(a) presents the temperature dependence of internal friction  $Q^{-1}$  for the as-supplied condition and after different charging times at the same charging current density. It can be seen that an increase of the charging time resulted in the changes in P1, P2 and P3 components. After 1 h of charging, only components P1 and P3 can be detected. Charging for 5 h gave rise to the component P2 along with existing P1 and P3 components. Further increase of the charging duration did not affect the heights of the components.

Fig. 3 shows the temperature dependencies of  $Q^{-1}$  for the hydrogen-charged F82H steel in the as-supplied condition for different oscillation frequencies. Components P2 and P3 decreased markedly with increasing frequency, while component P1 did not reveal noticeable changes taking into account the frequency shift of  $Q^{-1}$ .

Charging after cold rolling gave rise to a significantly higher hydrogen content in martensitic F82H steel (Table 2). It can be seen that the hydrogen content does not depend on the duration of the charging in the as-supplied condition. The obtained values are typical for this steel grade, for the specified heat treatment and the charging procedure [4]. However, the hydrogen content is significantly higher in the cold-deformed condition as compared to the as-supplied condition. Thus, the dislocations introduced during cold rolling are apparently effective traps for hydrogen at room temperature. On the

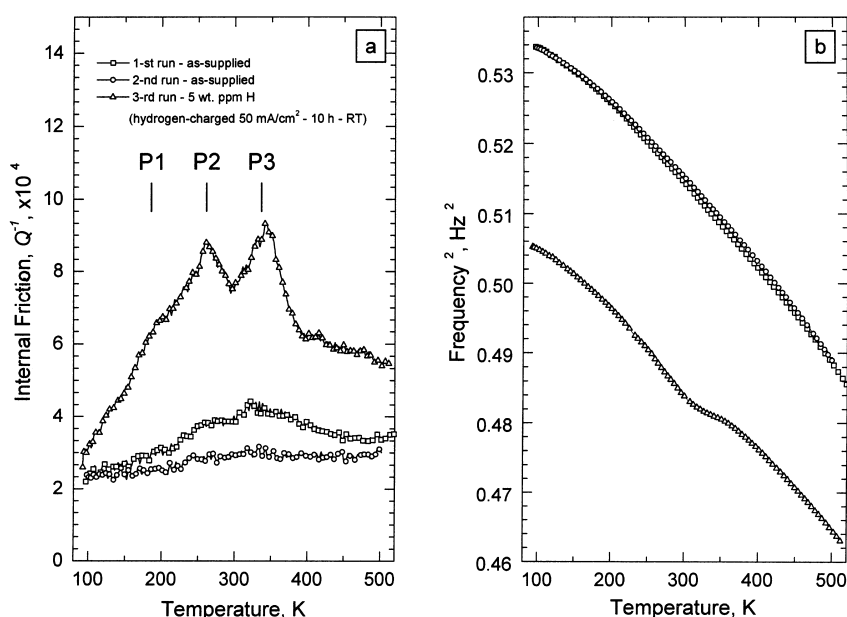


Fig. 1. Temperature dependencies of internal friction  $Q^{-1}$  (a) and the squared frequency of natural oscillations of the pendulum  $f^2$  (shear modulus) (b) in F82H steel in the as-supplied condition and after hydrogen charging with a current density of 50 mA/cm<sup>2</sup> for 10 h at room temperature.

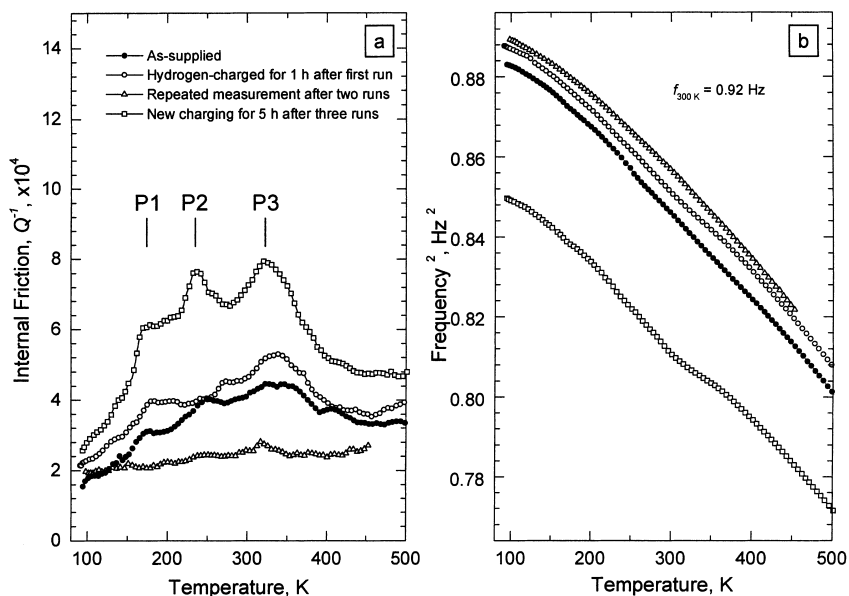


Fig. 2. Temperature dependencies of internal friction  $Q^{-1}$  (a) and the squared frequency of natural oscillations of the pendulum  $f^2$  (shear modulus) (b) of F82H steel in as-supplied condition after hydrogen charging with a current density of 50 mA/cm<sup>2</sup> at room temperature for different times.

other hand, F82H steel being deformed seems to be more sensitive to the surface damage during hydrogen charging, and surface cracks can be traps of gaseous hydrogen.

This gave rise to the study of the influence of cold work on IF in hydrogenated F82H steel. The temperature dependencies of  $Q^{-1}$  after cold working by tension and rolling followed by hydrogen charging at the identical conditions are given in Fig. 4. It can be seen that components P1 and P3 grew with increasing cold work

and component P2 seemed to change insignificantly. The IF spectrum for 10% cold-worked steel without hydrogen charging is also shown in Fig. 4. The absence of any distinct peaks assures that observed peaks P1, P2 and P3 are really caused by hydrogen.

Fig. 4 shows that peak P1 shifts to the lower temperatures with decreasing frequency as expected for a relaxation peak. It can be supposed that the origin of peak P1 and the hydrogen peak observed by Asano et al. in SUS 410 martensitic steel with the close chromium

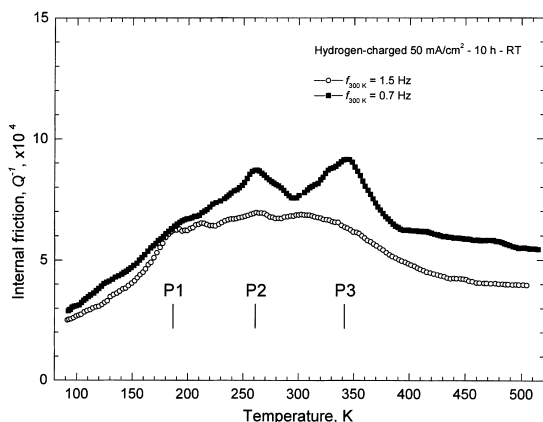


Fig. 3. Temperature dependencies of internal friction  $Q^{-1}$  at different oscillation frequencies in F82H steel in the as-supplied condition after hydrogen charging with a current density of 50 mA/cm<sup>2</sup> for 10 h at room temperature.

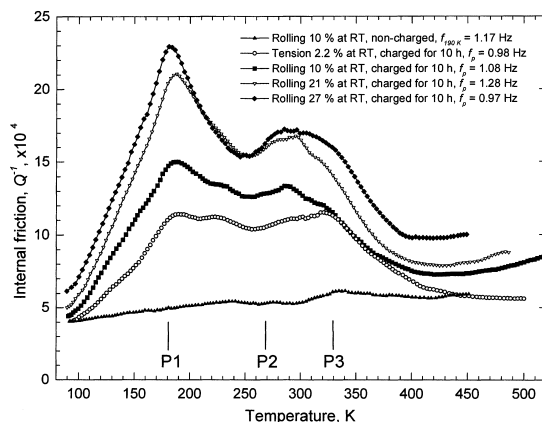


Fig. 4. Temperature dependencies of IF  $Q^{-1}$  in F82H steel after cold working by tension and rolling followed by hydrogen charging in the identical conditions with a current density of 50 mA/cm<sup>2</sup> for 10 h at room temperature.

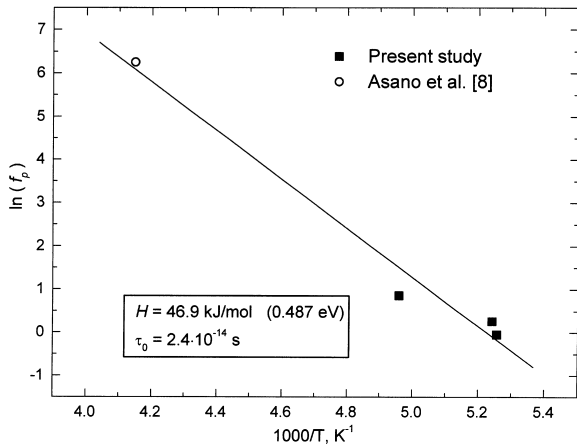


Fig. 5. Arrhenius plot for the calculation of activation parameters. Frequency at the temperature of the peak  $f_p = \omega/2\pi$ , where  $\omega$  is circular frequency of oscillation. The data of the present study are complemented with the data for the frequency of 500 Hz in SUS 410 steel by Asano et al. [8].

content (12 wt%) is the same [8]. Fig. 5 shows an Arrhenius plot built to evaluate the activation parameters, activation enthalpy  $H$  and pre-exponential factor  $\tau_0$  according to condition (1) (see below). Our data for the frequency of 1 Hz were complemented with the data of Asano et al. [8] for 500 Hz. The values of 46.9 kJ/mol and  $2.4 \times 10^{-14}$  s were obtained for the activation enthalpy  $H$  and the pre-exponential factor  $\tau_0$ , respectively.

Fig. 6 shows the temperature dependencies of internal friction  $Q^{-1}$  and frequency squared  $f^2$  for different heating rates. It can be seen that heating rate affects component P2, but not components P1 and P3. Component P2 increases with increasing heating rate. High-temperature IF background was higher for higher heating rate, as well. It should be noted that due to the thermal inertia of the equipment there exists a systematic error of the temperature measurement. The exact value of the error cannot be defined precisely. It can only be stated that the error is larger at higher temperatures and at higher heating rates. Thus, the apparent temperature positions of the peaks are higher than the true ones.

It should be pointed out that the temperature dependencies shown in Figs. 2–4 were obtained in the absence of external saturating magnetic field. To find out the contribution of magneto-elastic effects to the detected hydrogen peaks, a set of measurements was conducted in external magnetic field of 1500 Oe. The results are shown in Fig. 7. It can be seen that the magnetic field does not affect markedly the IF behaviour. The magnetic field suppresses slightly component P2 only. In other words, magneto-elastic effects in hydrogen-charged F82H steel give a small contribution to IF.

#### 4. Discussion

Internal friction of hydrogen-charged ferritic and martensitic steels with chromium was studied previously

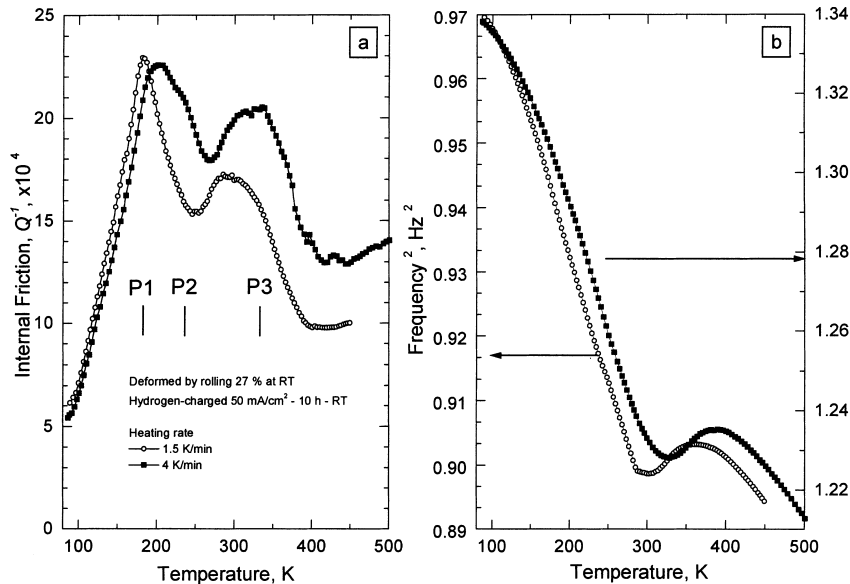


Fig. 6. Temperature dependencies of internal friction  $Q^{-1}$  (a) and frequency squared  $f^2$  (b) in F82H steel hydrogen-charged with a current density of 50 mA/cm<sup>2</sup> for 10 h at room temperature for two different heating rates.

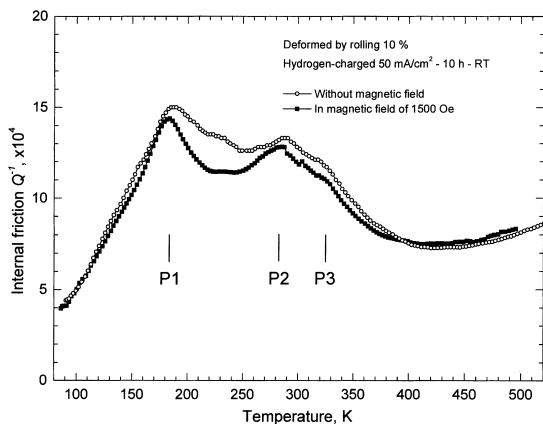


Fig. 7. Temperature dependencies of internal friction  $Q^{-1}$  in hydrogen-charged F82H steel with a current density of 50 mA/cm<sup>2</sup> for 10 h at room temperature with external magnetic field (approximately 1500 Oe) and without it.

by Asano et al. [8,9]. Relaxation internal friction peaks were detected on the temperature dependencies of the steels with different chromium content after cold working and hydrogen charging. In the kHz frequency range, the temperature position of the peak moves to the higher temperatures monotonously from 230 to 250 K with increasing chromium content from 6.3 to 27.9 wt%, respectively. In SUS 410 martensitic stainless steel (12.5 wt% Cr and 0.12 wt% C), the chemical composition of which is very close to the studied F82H steel, a peak was detected at 241 K for the frequency of 500 Hz [8].

Asano et al. [8] assumed the relaxation to be caused by the dislocation-hydrogen interaction and the detected peaks to be Snoek–Köster ones. Enthalpy of activation  $H$  and pre-exponential factor  $\tau_0$  of the relaxation time for the peaks were determined by the peak shift with the frequency  $\omega$  using the well-known expression [10]

$$\omega\tau = \omega\tau_0 \exp \frac{H}{RT_p} = 1, \quad (1)$$

where  $R$  is the universal gas constant,  $T_p$  the peak temperature. The obtained values of the activation enthalpy  $H$  were higher than 70 kJ/mol, and those of the pre-exponential factor  $\tau_0$  of the relaxation time were in the range of  $10^{-18}$ – $10^{-19}$  s. The binding energy of hydrogen atom to dislocations was calculated to be 32 to 45 kJ/mol depending on the chromium content [9]. The calculations were based on the Schoeck model of the Snoek–Köster relaxation [10], according to which the activation enthalpy is a simple sum of the activation enthalpy of a diffusion jump of a hydrogen atom,  $H_D$ , and the binding energy of the atom to dislocations,  $H_B$ ,  $H = H_D + H_B$ . The values of the activation enthalpy of diffusion  $H_D$  for the studied solid solution of chromium were taken from the literature.

Apparently, the IF peak detected in the present study in cold-worked and hydrogen-charged martensitic F82H steel containing 8 wt% Cr has the same origin as the above described relaxation peaks. At the same time, due to the significant difference of the frequency ranges (1 Hz in the present study and 500 Hz in the study of Asano et al.), there are some differences in the detailed structure of the observed peaks. In the low-frequency measurements of F82H steel, along with the relaxation low-temperature component P1, two non-relaxation components, P2 and P3, were detected. The temperature positions of these components do not change with varying frequency. Redistribution of hydrogen among different types of traps and its desorption from the sample are believed to be the reasons for the non-relaxation components P2 and P3. Transient IF peaks related to the hydrogen desorption were observed earlier in hydrogen-charged austenitic stainless steels [11] and Inconel-type nickel-base alloys [12]. It was shown that the amplitude of the transient peaks decreases with increasing frequency. Presumably, that is why Asano et al. did not observe these peaks in kHz frequency range.

In the present discussion, the main attention will be paid to the analysis of the relaxation component P1. The procedure of the deconvolution of the measured broad IF peak to P1, P2 and P3 components is problematic, since the apparent dependencies of internal friction on frequency, heating rate and temperature are needed for the transient components P2 and P3. Such dependencies are not available, yet, in the literature. Thus, only the temperature positions of the peaks can be determined.

Assuming that peak P1 in hydrogen-charged F82H steel has the same origin as the hydrogen peak in SUS 410 martensitic steel with similar chromium content, and using Arrhenius plot for the temperature positions of the peak (185 K at 1 Hz for F82H and 241 K at 500 Hz for SUS 410, respectively), the value of 46.9 kJ/mol can be obtained for the activation enthalpy of the peak. This value is noticeably lower than that mentioned in the studies of Asano et al. [9]. Pre-exponential value in this case is  $\tau_0 \approx 2.4 \times 10^{-14}$  s vs.  $10^{-18}$ – $10^{-19}$  s for SUS 410 steel [9].

There are two possible reasons for the relaxation peak P1. The first one assumes, like in the studies of Asano et al. [9], that the discovered peak is due to the interaction of dislocations with hydrogen atoms, i.e., it is the case of the Snoek–Köster relaxation. The alternative explanation takes into account the fact that, in an external field of elastic stresses, tetrahedral interstitial positions in the martensite lattice, initially equivalent with respect to their binding energy to a hydrogen atom, lose their energy degeneracy. This results in a diffusional redistribution of hydrogen atoms, which, in turn, leads to the relaxation IF process known as the Snoek relaxation [10].

The fact that P1 peak grows with increasing amount of cold work supports the Snoek–Köster mechanism of the relaxation. Such a behaviour is typical for the cold-work peaks in interstitial alloys [10]. Another typical feature known from numerous studies on carbon and nitrogen steels [10] is the dependence of the temperature position of the Snoek–Köster peak on the amount of cold work and on the interstitial content. The peak should move to the lower temperatures with increasing cold work and/or decreasing interstitial content. This behaviour is due to the decrease of the relaxation time with decreasing amount of interstitials trapped by dislocations.

The steels mentioned above had some initial average amount of carbon or nitrogen before deformation, which does not change with time. On contrast, electrolytic hydrogen-charging provides the same hydrogen content on dislocations independent of the amount of preliminary cold work, since the chemical potential of hydrogen rather than its average concentration is kept constant. Thus, the temperature position of the peak should not depend on the amount of cold work. Nevertheless, accurate analysis of the data given in Fig. 4 shows that for the identical conditions of hydrogen charging and with increasing amount of cold work the peak moves to lower temperatures. Asano et al. [8] did not observe any shifts in the peak positions in chromium-containing steels due to lower hydrogen content in the samples after preliminary desorption just before the measurements. Such shifts, however, take place in cold-worked chromium-free mild steel [8].

Thus, the interpretation of the peak P1 as the Snoek–Köster one meets the above mentioned contradictions. Additionally, the fresh dislocations produced by cold working have to be pinned with carbon atoms, since the solid solution content of carbon in F82H steel is, apparently, much higher than 0.004 wt% of the mild steel [8]. The pinning process by carbon atoms took inevitably place after the deformation during hydrogen-charging, which continued usually as long as 10 h.

The role of cold work in the P1 peak formation can have an alternative explanation based on Snoek mechanism of anelastic relaxation. One should take into consideration the fact that the distribution of alloying elements is inhomogeneous in the atomic scale in industrial martensitic steels of F82H type in the as-supplied condition. After the austenisation, the distribution of chromium and carbon atoms is not random and homogeneous, like in an ideal solid solution, but rather there are zones of enrichment and depletion with these elements [13]. Further redistribution takes place at tempering, although the inhomogeneous structure of the solid solution remains. Cold work, on contrary, leads to stirring of the solid solution due to the mass transfer by dislocations, and the distribution becomes more homogeneous [14].

In the BCT lattice of F82H steel, hydrogen atoms occupy tetrahedral interstitial sites (IS), like in the BCC lattice of iron. Volume density  $N_L$  of such sites is  $5.2 \times 10^{29} \text{ m}^{-3}$  [7]. The binding energy of a hydrogen atom is determined by its interaction with the nearest substitutional atoms, located in the vertices of the tetrahedron. The distribution of the latter depends, in turn, on the average chemical composition of the alloy and the type of atomic ordering in it. At temperature  $T$  the probability  $O_i$  of the site with a binding energy  $e_i$  to be filled is described with the Fermi statistics [15]:

$$O_i = \frac{1}{1 + \exp((e_i - \mu)/RT)}, \quad (2)$$

where chemical potential  $\mu$  should be determined from the conservation condition of the total amount of hydrogen atoms:

$$\sum_i \alpha_i O_i = C_0/N_L, \quad (3)$$

where  $C_0$  is the average volume concentration of hydrogen in alloy,  $\alpha_i$  the fraction of IS with the binding energy  $e_i$ . The sum is taken over all IS including those on dislocations, grain boundaries and other interfaces.

The distribution function  $a_i = a(e_i)$  describes the interaction of hydrogen with an alloy. The values of the binding energy  $e_i$  were estimated for different types of IS in iron and ferritic steels [7]. The activation energy of the lattice diffusion of hydrogen  $E_{id}$ , binding energy to deep traps  $e_t$  and the volume fraction of such traps,  $N_t = a(e_t)N_L$ , were determined for a set of martensitic chromium-containing steels, as well, by the method of thermodesorption analysis [7]. For F82H steel, the values of the parameters were determined to be 13.95, 55.94 and  $1.6 \times 10^{23} \text{ m}^{-3}$ , respectively. The values of the parameters for steels MANET and Grade 91 were obtained in an electrochemical study of the hydrogen permeability [16]. The values of  $N_t = 3.6 \times 10^{23} \text{ m}^{-3}$  for deep and irreversible traps,  $N_r = 1.2 \times 10^{28} \text{ m}^{-3}$  for reversible traps and  $e_r = 21.41 \text{ kJ/mol}$  were obtained for Grade 91 steel, close to F82H steel by its chemical composition and microstructure.

Grain boundaries and matrix-carbide particle interfaces are good examples of these deep traps in the studied F82H steel. They do not contribute to the hydrogen Snoek relaxation. The detected peak P1 seems then to be a hydrogen Snoek-like peak as a result of the transitions of hydrogen atoms between shallow reversible traps – tetrahedral IS in the lattice of F82H steel. The host tetrahedra are assumed to contain one or more chromium atoms, since the interaction energy of an isolated hydrogen-chromium pair in the BCC lattice of ferrite is estimated as 26.1 kJ/mol [17].

The binding energy of the reversible traps  $e_r$  can be calculated using the fact that for the Snoek-like relaxation the enthalpy of activation  $H \approx E_{id} + e_r$  and the

above given values of  $H$  and  $E_{\text{id}}$  for F82H steel. This value is between the binding energy of a tetrahedra with one and two chromium atoms. On the other hand, peak P1 is markedly broadened as compared to an elementary Debye peak, describing an ideal Snoek relaxation. Thus, there are reasons to assume that the peaks consist of several components, and the obtained value of  $\varepsilon_r$  should be considered as an apparent one. Finally, it should be noted that there is no information about the interactions of hydrogen with tungsten or carbon–tungsten compounds in F82H steel. Perhaps, the value  $\varepsilon_r \approx 32.95$  kJ/mol obtained in the present study corresponds to such kind of interactions.

Obviously, the values of  $a(\varepsilon_r)$  depend significantly on the thermal treatment and on the amount of cold work. Thermal treatment enhances decomposition of the solid solution to chromium-enriched and chromium-depleted regions resulting in a decrease of the fraction of IS tetrahedra with one or two chromium atoms. The subsequent deformation should increase parameter  $a(\varepsilon_r)$  up to the values typical for the ideal random solid solution. Thus, cold work increasing the value of  $a(\varepsilon_r)$  has to increase the height of peak P1.

From Eqs. (2) and (3) it follows that for a constant concentration hydrogen redistributes between different types of traps with temperature decreasing down to that of liquid nitrogen. Namely, filling of deeper traps grows at the expense of shallower ones. Hydrogen atoms leave dislocations with the binding energy of 20 to 30 kJ/mol [7] and become trapped by deeper lattice traps with  $\varepsilon_r \approx 32.95$  kJ/mol or grain boundary traps with  $\varepsilon_t \approx 55.94$  kJ/mol. At heating up during the IF measurement the redistribution process goes in the opposite direction, i.e., starting from some temperature hydrogen atoms escape from the reversible traps and occupy dislocations. Probably, it is this pinning of dislocations which is responsible for peak P2. At further heating, hydrogen leaves the sample by desorption. This transient process is accompanied with the changes in the elastic shear modulus, which results in non-relaxation peak P3. The detailed mechanism of the transient hydrogen peaks will be presented elsewhere.

## 5. Conclusions

1. A broad multicomponent internal friction peak was detected in F82H steel. The components are located at approximately 185 K (component P1), 260 K (component P2) and 340 K (component P3) at the frequency of oscillation of 1 Hz. Component P1 is a relaxation IF peak, while the other two components, P2 and P3, are related to the transient processes of hydrogen trapping by dislocations and hydrogen escape out of the sample, respectively.
2. Relaxation peak P1 is a Snoek-like peak and it is caused by the transition of hydrogen atoms between the energetically equivalent tetrahedral interstitial sites containing one or two chromium atoms in the vertices.
3. Cold work produces additional traps for hydrogen in F82H steel, which increases the absorption capability of the solid solution in electrolytic hydrogen charging.
4. The binding enthalpy of a hydrogen atom to the lattice trap equals to 32.95 kJ/mol, according to the internal friction results. The fraction of such traps depends on the heat treatment and the amount of cold work of martensitic F82H steel.

## Acknowledgements

This study is a contribution to the Finnish Fusion Research Program (FFUSION) as a part of the Underlying Technology Program of TEKES (Technology Development Centre of Finland).

## References

- [1] R. Boler, C.B.A. Forty, G.J. Butterworth, in: An Initial Examination of the Likely Effect of Hydrogen on Low Activation Martensitic Stainless Steels, AEA Report, AEA FUS 164, February 1992.
- [2] S.M. Myers, M.I. Baskes, H.K. Birnbaum, J.W. Corbett, G.G. Delo, S.K. Estreicher, E.E. Haller, P. Jena, N.M. Johnson, R. Kirchheim, S.J. Pearton, M.J. Stavola, *Rev. Mod. Phys.* 64 (2) (1992) 559.
- [3] G. Benamatti, A. Donato, A. Solina, R. Valentini, in: K. Hirschbach, W. Maurer, J.E. Vetter (Eds.), *Fusion Technology 1994*, Elsevier Science, Amsterdam, 1995 p. 407.
- [4] T.A. Lechtenberg, W.M. Garrison, J.M. Hyzak, in: J.W. Davis, D.J. Michel (Eds.), *Proceedings of the Topical Conference on Ferritic Alloys for Use in Nuclear Energy Technology*, TMS-AIME, Warrendale, PA, 1984, p. 365.
- [5] A. Kimura, H. Kagano, M. Narui, *J. Nucl. Mater.* 179–181 (1991) 737.
- [6] A. Turnbull, M.W. Carrol, D.H. Ferriss, *Acta Metall.* 37 (1989) 2039.
- [7] E. Serra, A. Perujo, G. Benamatti, *J. Nucl. Mater.* 245 (1997) 108.
- [8] S. Asano, A. Kimura, R. Otsuka, *Scr. Metall.* 12 (1978) 805.
- [9] S. Asano, M. Shibata, *Scr. Metall.* 16 (1982) 1171.
- [10] A.S. Nowick, B.S. Berry, *Anelastic Relaxation in Crystalline Solids*, Academic Press, New York, 1972, p. 677.
- [11] V.G. Gavriljuk, H. Hänninen, S. Smouk, A.V. Tarasenko, K. Ullakko, *Metall. Trans.* 27A (1996) 1815.
- [12] P. Aaltonen, Yu. Jagodzinski, A. Tarasenko, S. Smouk, H. Hänninen, *Philos. Mag.* 78 (4) (1998) 979.
- [13] R. Coppola, P. Gondini, R. Montanari, *J. Nucl. Mater.* 206 (1993) 360.



- [14] M. Grujicic, W.S. Owen, *Acta Metall. Mater.* 43 (1995) 4201.
- [15] R. Kirchheim, F. Sommer, G. Schluckebier, *Acta Metall.* 30 (1982) 1059.
- [16] G. Benamatti, A. Donato, A. Solina, R. Valentini, S. Lanza, *J. Nucl. Mater.* 212–215 (1994) 1401.
- [17] A.I. Shirley, C.K. Hall, *Scr. Metall.* 17 (1983) 1003.