HYDROGEN DIAGNOSTICS OF STRUCTURAL CHANGES IN STEEL 18Cr10NiTi SUBJECTED TO LOW-TEMPERATURE EXTRUSION

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Introduction

One of the application aspects of thermally activated desorption spectrometry is the analysis of hydrogen desorption kinetics that enables, on the basis of the measured spectra, the evaluation of thermodynamic parameters of the structural condition of the material, viz., the number of desorption peaks, their temperature ranges, activation energy. The correlation has been established [1, 2] between the peaks in thermodesorption spectra and the phase transformations in the Ti-D system such as TiD_2 deuteride decay, the phase transition of titanium (α-Ti $\Leftrightarrow$ β-Ti), the decomposition of deuterium solid solution in β-Ti. The experiments [3-5] have revealed the influence of existing interstitial impurities (C, H, O, He) on the temperature ranges of hydrogen retention in 18Cr10NiTi austenitic stainless steel, and hence on the structural changes.

The deformation of 18Cr10NiTi austenitic stainless steel at cryogenic temperatures under the conditions close to hydrostatic compression (quasi-hydrostatic extrusion) at a high pressure level provides fine-dispersion martensite formation in the steel, this bringing to a substantial gain in the mechanical characteristics of steel, namely, to the rise of yield and breaking points [6, 7].

This paper presents the results of studies aimed at investigating the degree of influence of 18Cr10NiTi austenitic stainless steel cryodeformation on the hydrogen desorption kinetics in order to estimate structural changes of the material.

Experimental procedure

For experiments, we have used cylindrical steel 18Cr10NiTi work parts that underwent a homogenizing annealing through heating up to 1320 K, holding at this temperature for an hour and quenching in water. The samples prepared in this way were then subjected to quasihydroextrusion at ~78 K in the range of percent reduction δ = 16% and 44%. After an unforced warming up to room temperature the deformed samples were placed into a vacuum chamber, where they were fixed on the substrates (made from the same steel of thickness δ ~0.3 mm) that served as heaters. Deuterium was implanted by exposing the samples to the D_2^+ ion beam of energy 24 keV and a current density of ~5 $\mu A/cm^2$ at a temperature of ~ 140 K to doses of 1×10^{16} … 1×10^{17} D/cm^2. The irradiated samples were heated up to temperatures of 800 … 1300 K at an average rate of ~ 8 K/s with a simultaneous mass-spectrometer detection of deuterium desorption. In the process of heating the temperature was measured with the help of a chromel-alumel thermocouple fixed to the sample.

Results and discussion

Figure 1 shows the most characteristic deuterium thermodesorption (TD) spectra obtained in the present experiments. As it can be seen from the figure, up to a dose of ~1×10^{17} cm^{-2} the spectrum exhibits one peak with the maximum temperature T_m~410 K.

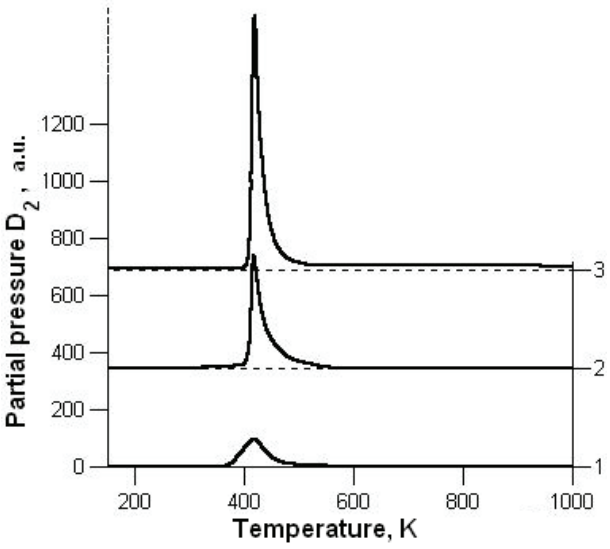

Fig. 1. Thermodesorption spectra of deuterium implanted into 18Cr10NiTi steel at T_{irr}~140 K measured at exposure doses: 1 – $1\times10^{16}$$cm^{-2}$; 2 – $3\times10^{16}$$cm^{-2}$; 3 – $1\times10^{17}$$cm^{-2}$.

Having chosen a low hydrogen implantation dose as a test dose (hydrogen implantation dose

that corresponds to the phase of hydrogen solid solution in 18Cr10NiTi steel, and therefore, does not affect the metal structure), it appears possible to estimate from the TD spectra the structural changes in the 18Cr10NiTi steel that underwent low-temperature extrusion. However, the use of low doses of implanted hydrogen in experiments involves difficulties in the interpretation of the experimental results, caused by the presence of hydrogen in the vacuum chamber as a residual gas component. In view of this, deuterium as the hydrogen isotope was used in the experiments.

The TD spectra of deuterium implanted into 18Cr10NiTi steel samples subjected to homogenizing annealing and quasihydroextrusion deformation at ~78 K are presented in Fig. 2. As is evident from the figure, the low-temperature quasihydroextrusion of annealed 18Cr10NiTi steel samples leads to a qualitative change in the TD spectrum of implanted deuterium. The evolution of the gas release spectrum is observed, depending on the reduction ratio of the samples. At δ=16%, at least two peaks of deuterium desorption intensity with the maxima at T_m~350 K and T_m ~520 K (curve 2) manifest themselves. The increase in the reduction ratio up to δ=44% results in a single-peak structure of the gas release spectrum (curve 3). The present data are in good agreement with the results of earlier studies on phase transformations during deformation, which occur by the $\gamma \Rightarrow \varepsilon \Rightarrow \alpha$, scheme, where γ denotes the austenite with the ***fcc*** lattice, α is the martensite with the ***bcc*** lattice, and ε is the martensite with the ***hcp*** lattice [7].

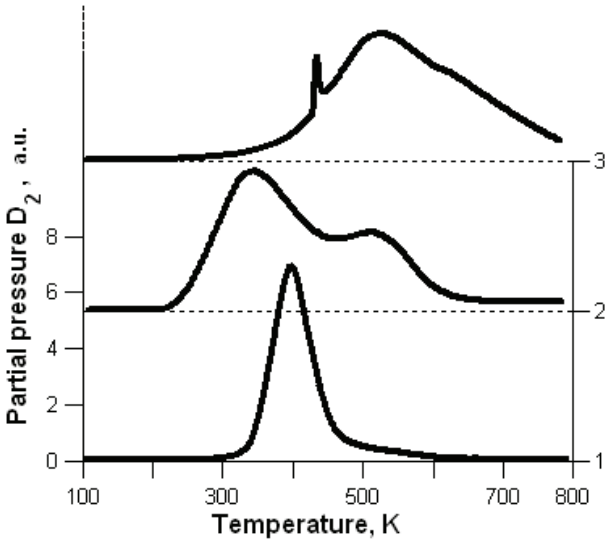

Fig. 2. TD spectra of deuterium implanted into 18Cr10NiTi steel samples subjected to quasihydroextrusion deformation at 78 K with percent reductions δ =16% (curve 2) and 44% (curve 3). For comparison, curve 1 shows the spectrum of the initial steel sample. The exposure dose for all the samples is 5×10^{16} D/cm^2.

The hydrogen diagnostics points obviously to the presence of two martensite phases, one of which is the transition phase (ε - martensite, T_m~350 K), which is present in the sample with δ=16%. Between the peaks, there is presumably an unresolved peak with T_m~400 K, specified by the presence of the residual γ – austenite. The samples deformed at δ=44% are characterized by a complete transformation of austenite into martensite. Note that the peaks in the spectra are broad, this confirming a high degree of dispersion of the resulting phases.

The hydrogen diagnostics should be reckoned among promising directions of studies on structural states of materials.

References

1. Neklyudov I.M., Morozov A.N., Kulish V.G. Temperature ranges of hydride phase stability of the TiD system. (In Russian). Materialovedenie 2005; 11: 45-46.

2. Neklyudov I.M., Morozov A.N., Voyevodin V.N., Kulish V.G.. Kinetics of structural transformations in titanium during deuterium ion irradiation and post-implantation annealing. (In Russian). Al'ternativnaya ehnergetika i ehkologiya 2004; No 5(13): 5-11.

3. Neklyudov I.M., Morozov A.N., Kulish V.G., Zhurba V.I. The effect of interstitial impurities on temperature ranges of deuterium retention in austenitic stainless steel 18Cr10NiTi. (In Russian). Hydrogen economics and hydrogen treatment of materials. Proc. of Int. Conf. "BOM-2007". Donetsk, 2007; 763-767.

4. Neklyudov I.M., Morozov A.N., Kulish V.G., Zhurba V.I., Galitsky A.G. Retention of hydrogen isotopes in 18Cr10NiTi steel implanted with helium ions. (In Russian). Voprosy Atomnoj Nauki i Tekhniki. Series: Termoyadernyi sintez 2008; 2: 41-46.

5. Neklyudov I.M., Morozov A.N., Kulish V.G., Zhurba V.I. The influence of interstitial impurities on temperature ranges of deuterium retention in austenitic stainless steel. J. of Nuclear Materials 2009; 386-388: 658-660.

6. Khaimovich P.A. Cryodeformation of metals under uniform pressure. (In Russian). Voprosy Atomnoj Nauki i Tekhniki. Series: Fizika radiatsionnykh povrezhdenij i radiatsionnoe materialovedenie. 2006; 4(89): 28-34.

7. Bryk V.V., Neklyudov I.M., Sokolenko V.I., et. al. Low-temperature quasihydroextrusion as a way of strengthening structural materials. (In Russian). Metallofiz. Noveishie Tekhnol. 2005; 27(4): 551-562.