Deuterium concentration's in austenitic stainless steel by deuterium irradiation. Effects temperature irradiation

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Abstract. Using the deuterium thermodesorption spectra from the 18Cr10NiTi steel samples exposed to different doses, we have plotted the total amount of deuterium desorbed from the sample as a function of the radiation dose and the irradiation temperatures. *Temperature 100 K.* The maximum attainable concentration of deuterium in steel is C=1 (at.D/at.met.=1/1). At C≥0.5, two hydride phases are formed in the steel, the decay temperatures of which are 240 K and 275 K. The hydride phases are formed in the bcc structure resulting from the martensitic structural transformation in steel [1]. *Temperature 295 K.* The medium-dose region is characterized by radiation induced action on the steel in the presence of hydrogen. The process results in the formation of the energy-stable crystalline nanostructure of steel, having a developed network of intercrystalline boundaries. The basis for this developed network of intercrystalline boundaries is provided by the amorphous state. The total concentration of the accumulated deuterium in the region of medium implantation doses makes 7 to 8 at.% [2]. *Temperature 380; 420; 600 and 900 K*. In a deuterium thermodesorption spectra the extended area of deuterium desorption in a range of temperatures 450-900 K is observed, caused by formation of local structure in a radiation induced layer. Formation of local structure can be caused by a segregation of a steel component in the course of deuterium implantation (radiation induced local structure at deuterium presence).The total concentration of the accumulated deuterium in the region of medium implantation doses makes 1 to 3 at.%.

1. Introduction

The investigation into regularities of hydrogen interaction with metals and alloys over a wide range of temperatures and pressures still remains a currently central problem in material physics from both the scientific and practical standpoints. The hydrogen accumulation in structural and functional materials is an extremely hazardous phenomenon, which leads to hydrogen degradation of materials and to possible unforeseen equipment failures. The degradation of materials increases due to hydrogen interaction with the whole range of crystal structure imperfections of solids such as interstitial and substitutional impurities, vacancies and their complexes, dislocations and their pileups, subgrain and grain boundaries, and phase components. Stainless steel is one of the most useful classes of engineering materials. For example, austenitic steel is used for manufacturing vessel internals of fission-type reactors. A wide use of austenitic stainless steels as structural fission reactor elements calls for a detailed knowledge of their behavior under conditions of radiation influence, accumulation of gas impurities (hydrogen isotopes, above all). Hydrogenation of the face-centered cubic (fcc) iron-based alloys, which constitute an array of austenitic stainless steels, can cause phase transformations: fcc (γ)→bcc (α*) and fcc (γ)→hcp (ε) [3][4]. The hydrogen-induced phases are sometimes considered as pseudohydrides. It is generally assumed that the role of hydrogen consists in creation of a particular

stress state that triggers the phase transformation. Morozov and co-workers [1] showed the steel surface layer exposed to deuterium ions at Tirr.= 100 K to doses above 8.2 ×10^{17} D/cm^2, exhibits austenite, α-martensite needles.

At room temperature, deuterium has a high diffusive mobility, the surface open to diffusion, and thereby is intensively desorbed from austenitic steel during its implantation. As is known, at room temperature, nearly all the defects formed in the course of irradiation, and above all, the Frenkel pairs are recombining [5]. In the process of deuterium ion implantation in the steel being at temperature 295 K, there occurs the formation of a great number of chaotically distributed dislocations, small precipitates, separate ordered segregations, which provide the basis for the occurrence of a nanocrystalline microstructure at a dose of 1.68 × 10^{18} D/cm^2 [2].

The present paper presents the results from studies the TDS deuterium of the sample irradiated to different doses, we have plotted the total concentration of deuterium, desorbed from the specimen as a function of radiation dose and temperature irradiation.

2. Experimental procedure

Austenitic steel 12Cr18Ni10Ti (0.12 wt.%C) and 08Cr18Ni10Ti (0.08 wt.%C) samples were investigated. The samples were homogenized at 1,350 K for 30 min in a vacuum of 5 × 10^{-5} Pa. Deuterium introduction into the samples was realized through implantation of 24 keV D_2^+ ions at a current density of 2 to 5 μA/cm^2, with doses ranging from 1×10^{15} to 4×10^{18} at.D/cm^2; the irradiation temperatures were T ≥ RT. The implanted samples were subjected to heating immediately after the ion beam switching-off at a rate of 3.5 – 5 K/s down to a temperature of 1700 K, with simultaneous registration of D_2^+ ions ion desorption spectrum (4 amu).

3. Results and discussion

Using the deuterium thermodesorption spectra from the 18Cr10NiTi steel samples exposed to different doses, we have plotted the total amount of deuterium C(F) desorbed from the sample as a function of the radiation dose F and the irradiation temperatures.

3.1 Deuterium Thermodesorption Spectra at Temperatures 380; 420; 600 and 900 K

The deuterium TD spectrum of steel 12Cr18Ni10Ti irradiated at 380 K (see Figure 1, curve b) exhibits only a low-intensity, wide temperature-range region of deuterium desorption, which begins practically from the irradiation temperature and extends up to ~ 1200 K. Here, it is clearly seen that this region is composed of three peaks with the maximum temperatures of 480, 700 and 1020 K. The presence of these peaks in the TD spectrum testifies that in the process of deuterium implantation in the steel 12Cr18Ni10Ti local structural compositions from steel components were formed. We have made this conclusion on the basis of the data obtained in ref. [32-33], where it was demonstrated that the ion implantation of nitrogen, oxygen, carbon, helium could lead to the formation of such local structures as nitrides, carbides, the presence of which in the deuterium TD spectra manifests itself in the form of

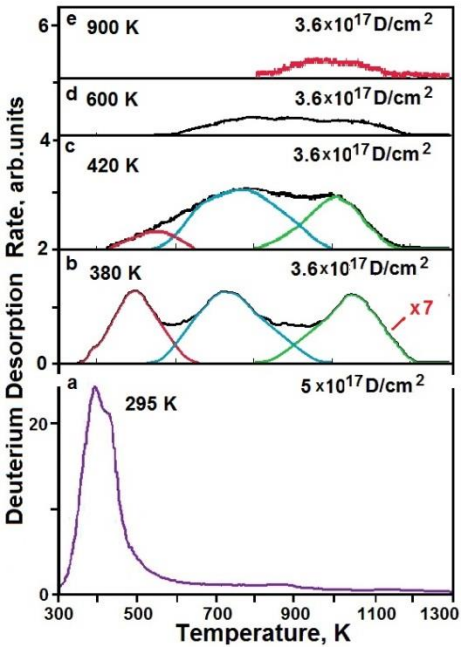

Figure 1. Deuterium thermal desorption spectra of steel 12Cr18Ni10Ti samples, irradiation with deuterium at temperatures of 295 K [1], 380, 420, 600, 900 K to a dose of 3.6×10^{17} D/cm^2

occurrence of additional temperature ranges of deuterium desorption (hydrogen diagnostics). With further increase in the irradiation temperature, both the amount of retained deuterium and the temperature range of deuterium desorption get reduced.
It is assumed that carbon may be one of the steel components capable of stimulating the local structure formation. This assumption is supported by the data on the carbon behavior in nickel, reported in ref [6]. It has been demonstrated there that on heating of nickel, which does not form chemical compounds with carbon, the latter diffuses to the crystallite boundaries. It is reasonable to expect that the carbon behavior in the austenitic stainless steel is similar to the behavior of carbon in nickel. On this basis it can be suggested that the irradiation of steel with deuterium might stimulate the formation of local structures from steel components and carbon.

3.2 Effect of carbon concentration in steel on temperature ranges of deuterium retenthion retention

For estimating the possibility of local structure formation from steel components and carbon, studies were made into deuterium TD spectra from steels having different carbon concentrations: 12Cr18Ni10Ti (0.12 wt.%C) and 08Cr18Ni10Ti (0.08 wt.%C). The deuterium TD spectra from these steels, irradiated at temperatures of 295 and 900 K, are presented in Figure 2. As may be seen from the figures, in the spectrum from the steel with a lower carbon concentration (0.08 wt. % C) there is no highest-temperature spectral region, i.e., there is no additional peak with the maximum temperature of 1020 K. At the same time, in the case of 12Cr18Ni10Ti steel irradiation with a higher carbon concentration (0.12 wt. %C), the local structure formation takes place, which manifests itself in the occurrence of the additional peak with the maximum temperature of 1020 K. Relying on the data obtained, we have concluded that the carbon present in the 12Cr18Ni10Ti steel stimulates the steel components segregation and the local structure formation.

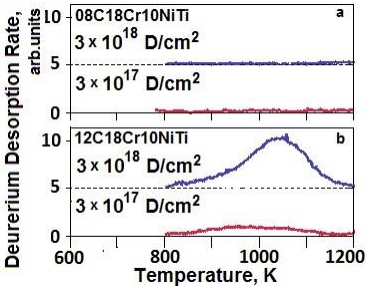

Figure 2. Deuterium thermal desorption spectra from steel samples: a) 08Cr18Ni10Ti, b) 12Cr18Ni10Ti, irradiation at Tirr=900 K

3.3 The amount of retained deuterium as a function of temperature and radiation dose

Using the deuterium thermodesorption spectra from the Cr18Ni10Ti steel samples exposed to different doses, we have plotted the total amount of deuterium C(F) desorbed from the sample as a function of the radiation dose F. This function is presented in Figures 3a and 3b. The total amount of the desorbed deuterium was determined from the area under the gas release curve.
The concentrations of implanted deuterium were estimated with consideration for both the quantity of metal atoms in the implantation layer and the amount of desorbed deuterium. At that, the implanted deuterium was assumed to have low diffusion mobility in the steel cooled down to Tirr=100 K, and practically all its amount be in the implantation layer. The calculation data have shown that the Cr18Ni10Ti steel saturation with deuterium is attained at the deuterium concentration C= 1.0 at.D/at.met. [1].

At Tirr= 295 K (RT), in the range of low implantation doses, the concentration of accumulated deuterium varies between 2.5 to 3 at.%. On completion of deuterium solid-solution formation in steel, the newly implanted deuterium atoms give impetus to further structural transformations, which are due to both the radiation action and the increasing concentration of implanted deuterium. In this case, the amount of retained deuterium increases at the expense of growing intensity of the high-temperature region of the spectrum with the peak temperatures 440 and 500 K. At a dose of 8×10^{17} D/cm^2, the accumulation of retained deuterium at the peak with Tm = 440 K practically fully ceases. The deuterium concentration in this phase state remains practically unchanged during further deuterium ion implantation, and is estimated to be 7–8 at.%. The irradiation temperature increase is accompanied by

the reduction in the retained deuterium concentration down to ~ 1 at.% at temperatures between 420 to 600 K.

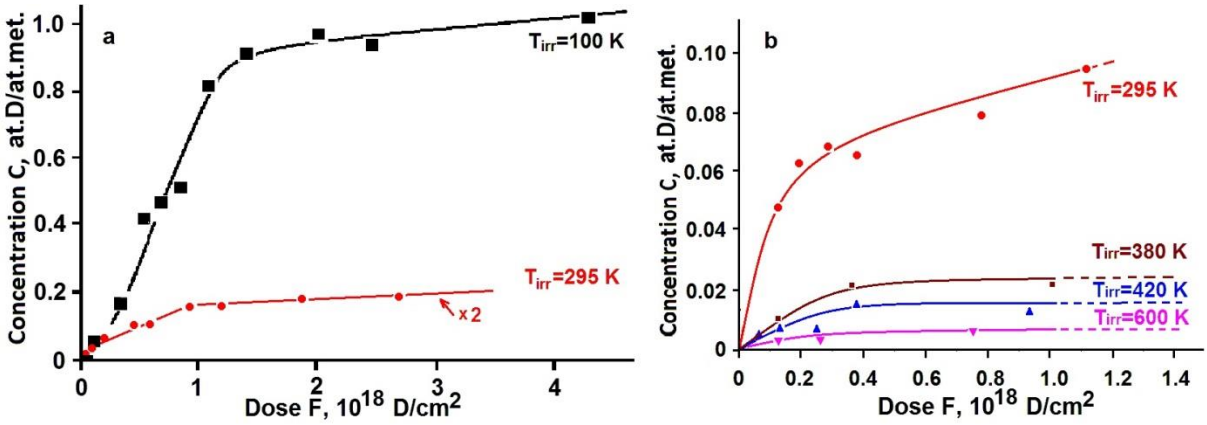

Figure 3. Desorbed deuterium concentration versus irradiation dose for the Cr18Ni10Ti steel implanted at different temperatures

4. Conclusions

Temperature 100 K. The maximum attainable concentration of deuterium in steel is C=1 (at.D/at.met.=1/1). At C≥0.5, two hydride phases are formed in the steel, the decay temperatures of which are 240 K and 275 K [1].

Temperature 295 K. The medium-dose region is characterized by radiation induced action on the steel in the presence of hydrogen. The process results in the formation of the energy-stable crystalline nanostructure of steel, having a developed network of intercrystalline boundaries. The basis for this developed network of intercrystalline boundaries is provided by the amorphous state. The total concentration of the accumulated deuterium in the region of medium implantation doses makes 7 to 8 at.% [2].

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