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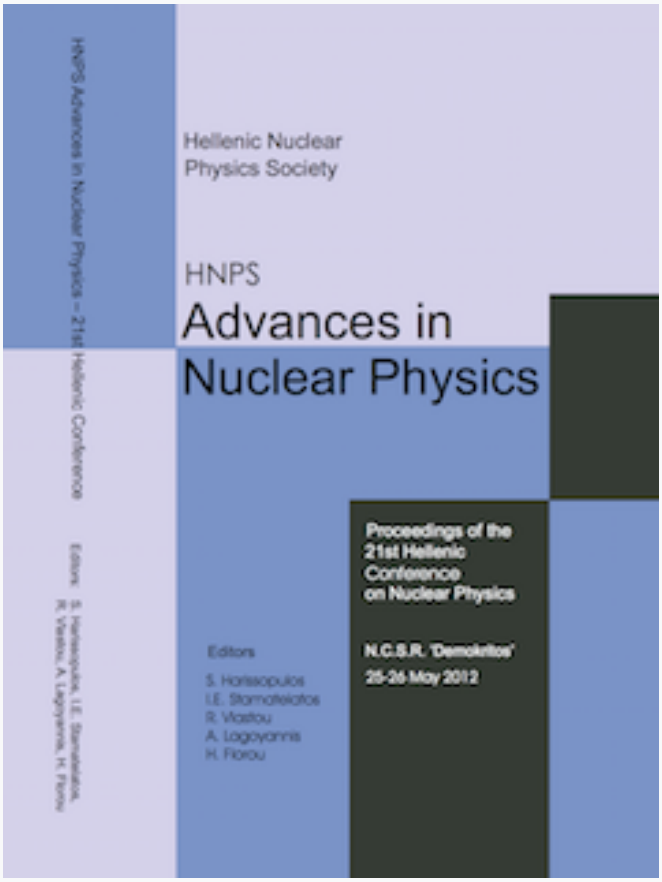

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Radiation damage studies of Fe-Cr alloys for Fusion applications using ion beams

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Abstract

Ferritic-martensitic steels are considered as prime candidates for the first wall and blanket structural materials in future Fusion Power Plants. They are based on Fe-Cr alloys with Cr concentration up to 15%. Therefore, as a first step towards understanding the irradiation behaviour of these steels, the study of pure Fe-Cr alloys as a model system plays an important role. The flexibility offered by ion beams in accurately defining irradiation conditions, allows us to perform controlled experiments that will aid in the validation of recent theories of radiation damage.

Radiation damage studies of Fe-Cr alloys were performed at the TANDEM accelerator of NCSR "Demokritos" by using a 5MeV proton beam directly on the sample and reaching fluences of ~10^{16} protons/cm^2. A specialized irradiation facility has been developed at the TANDEM accelerator with good control of irradiation temperature and with the additional capability of measuring the electrical resistivity of the sample *in-situ* during irradiation, to directly asses the radiation damage evolution. Proton irradiations were carried out from cryogenic temperatures of 40K up to 400K. In alloys under irradiation, apart from the lattice damage created by the irradiating particles, the large amount of energy imparted locally to atomic sites may lead to a redistribution of solutes. It is expected that at cryogenic temperature solute redistribution will be greatly suppressed, since atomic movement is completely "frozen". Comparing the results of low and high temperature irradiations enables us to distinguish between the two sources of radiation induced changes to the alloy.

Keyword: Fusion, Fe-Cr alloys, ion irradiation, electrical resistivity

1. Introduction

Ferritic-martensitic steels, based on iron-rich Fe-Cr alloys, receive special interest as structural materials for the first wall and blanket in future fusion power plant reactors [1], mostly due to their resistance to swelling, the main factor of radiation damage in austenitic stainless steels. However some important engineering properties, such as embrittlement and high-temperature strength, remain open issues as ferritic steels rapidly lose strength at temperatures higher than 550^{o}C. Therefore understanding how these materials behave in the fast neutron-environment of a fusion plant and what the degradation rate of their key properties is, can be described as one of the greatest materials science challenges towards realization of fusion power generation.

A number of studies have been performed to quantify and qualify the detrimental consequences of radiation caused by fast/thermal neutrons [2-4], charged particles [5, 6] and ions [7]. The consequences of radiation are referred to as radiation damage. The radiation damage event is defined as the transfer of energy from an incident particle to the solid and the resulting distribution of target atoms after completion. The

result of a radiation damage event is the creation of a collection of point defects (vacancies and interstitials) and clusters of these defects in the crystal lattice. Migration of the point defects and defect clusters and additional clustering or dissolution of the clusters, are classified also as radiation damage effects. In addition, changes in the microstructure of the alloys during service are governed by the kinetics of defects produced by irradiation. Such microstructural changes are of crucial importance since they may lead to a degradation of the macroscopic alloy properties and, finally, to failure.

In this contribution we present results for the radiation damage evolution during proton irradiation of concentrated $Fe_{1-c}Cr_c$ alloys (c=0.00, 0.05, 0.10, 0.15) at low (40 K) and high (400 K) temperature. *In-situ* measurements of the electrical resistivity are used for real-time observation of the irradiation induced modifications to the sample. Electrical resistivity is extremely sensitive to the presence of irradiation defects and the relative simplicity of its measurement renders it a useful tool for radiation damage studies. In section 2 some experimental details are discussed briefly. Then, the results are presented, analyzed and discussed in section 3 and in the last section we give the conclusions.

2. Experimental

Fe-Cr model alloys with nominal concentrations of 5.8, 10.8 and 15 at. % Cr and pure Fe reference material were obtained from EFDA (European Fusion Development Agreement). The materials were cold rolled to foils of 50 μm thickness and specimens were spark-cut from these foils. After chemical polishing for the removal of any possible surface contamination the specimens were annealed for 24 hours at 800ºC under vacuum and then quickly removed from the furnace. The specimens were loaded in the irradiation facility and instrumented with current and voltage leads for resistivity measurements. Uniform heating is accomplished by two heaters on either side of the sample strip. Two type K thermocouples are attached at the sample periphery for direct measurement of temperature and for checking the temperature homogeneity.

After loading, the samples were cooled to a base temperature of 15 K and their residual resistivity was measured. Irradiation was performed with 5 MeV protons at a flux of $\sim 5\times10^{11}$ $cm^{-2}s^{-1}$ and to a maximum total fluence of $\sim 10^{16}$ cm^{-2}. At this energy the proton range in Fe and Fe-Cr is 80 μm as calculated by the SRIM code. The range does not depend appreciably on Cr presence. Thus, the proton beam fully penetrates the 50 μm thick specimens and the induced material modification is distributed homogeneously throughout the specimen volume.

3. Results and discussion

The resistivity changes recorded during proton irradiation of the pure Fe specimen are presented in Fig. 1. At the irradiation temperature of 40 K a linear increase of the resistivity $\Delta\rho$ is observed as a function of proton fluence. At this temperature the defects created by the irradiation are immobile. As a result the resistivity constantly increases due to the build up of the defect concentration in the lattice. The rate of resistivity increase is $\sim 3.9\times10^{-17}$ μΩ-cm^3. In contrast, during high temperature irradiation, there is no detectable resistivity change. The resistivity of the sample remains constant with irradiation dose, within experimental resolution.

In Fig. 2, the results for a Fe-5 at. %Cr alloy under the same experimental conditions can be seen. The irradiation at low temperature results in a linear increase

of the resistivity, similar to that observed in pure Fe. The rate of resistivity increase in this case is $\sim 2.2\times10^{-16}$ μΩ-cm^3. This is true for all studied Cr concentrations up to 15 at.%. On the other hand, at 400 K, the behavior is completely different in the alloy compared to pure Fe, and differs also with Cr concentration. Fig.2 shows that for the 5 at.% alloy, during high temperature irradiation, the resistivity first increases, but exhibits saturation and finally reaches a plateau.

In Fig. 3, the results for Fe-10at.%Cr and Fe-15at.%Cr are compared to Fe-5at.%Cr, under irradiation at 400 K. The observed peculiar behavior for Fe-5 at.%Cr, not only remains, but appears inversion at higher Cr concentrations. At $C_{Cr} \geq 10$ at. % the resistivity decreases with irradiation and reaches a plateau which depends on concentration. This saturation has a characteristic time between 40 – 80 minutes, also concentration dependent (resistivity curves vs fluence can be expressed by exponential increase-decrease functions).

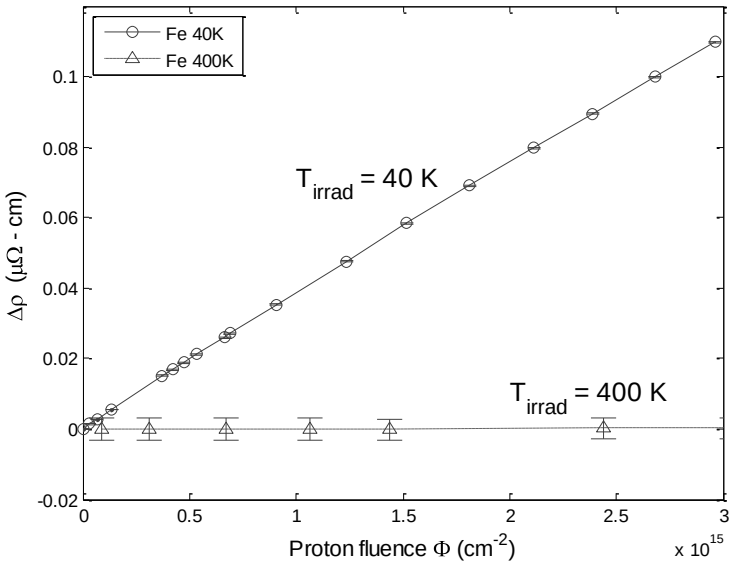

Fig. 1. Change of the electrical resistivity of pure Fe as a function of fluence during 5MeV proton irradiation at low and high temperature.

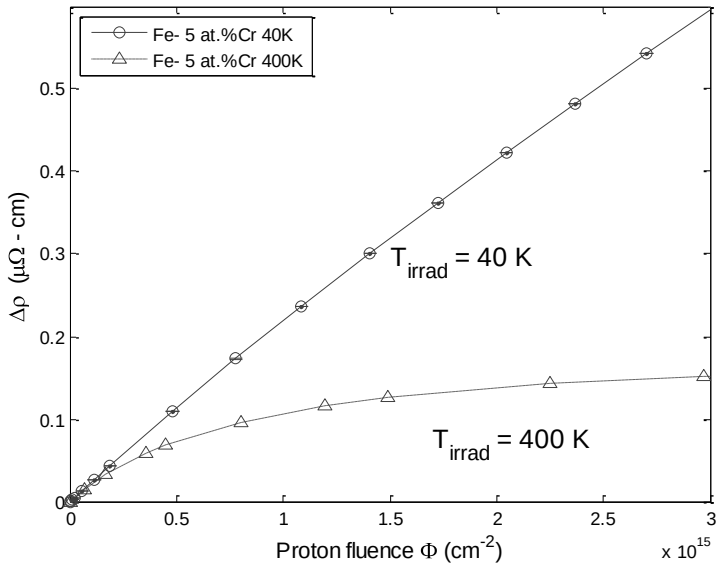

Fig. 2. Change of the electrical resistivity of a Fe-5% Cr alloy as a function of fluence during 5MeV proton irradiation at low and high temperature.

The results show that irradiation at cryogenic *T*, causes a constant rate of defect production both in pure Fe and in the Fe-Cr alloy. These defects are mainly interstitial atoms and vacancies formed as lattice atoms are knocked out of their lattice positions by the impinging protons. There is no reason to believe that defects are produced at a significantly higher rate in the alloy than in pure Fe, since Fe and Cr have very similar atomic masses and thus behave similarly during scattering with the energetic beam particles. Thus, the larger slope of the resistivity vs dose curve must be due to a difference of the resistivity per unit defect concentration, which is apparently higher in the alloy. The physical origin of this effect is not clear at this moment.

The absence of detectable changes in the resistivity of pure Fe at 400 K shows that at this temperature the irradiation defects migrate rapidly and finally annihilate, either mutually or at defect sinks in the sample. Looking at the fundamentally different behavior of the Fe-5 at.%Cr alloy at the same irradiation temperature, shown in Fig. 2, one could assume that defect migration is much slower in the alloy than in pure Fe. However, we know from previous radiation damage recovery experiments on Fe-Cr [8] that this is not the case. Thus, the observed changes for the alloys, in combination with the sign reversal of the change in resistivity, are attributed to a redistribution of Cr solute atoms in the alloy, which is facilitated by the energy imparted to lattice atoms by the irradiating particles. This means that the atomic diffusion constant of the alloys is effectively enhanced by the irradiation. In addition, the enhanced diffusion allows the system to re-order at a new equilibrium configuration that corresponds to the irradiation temperature. Atomic ordering in alloys can be described by the Cowley-Warren [9] short range order (SRO) parameters, α_i, which quantify the tendency of Fe and Cr atoms to form pairs or not. Our results regarding the behavior at 400 K during irradiation indicate that α_i changes sign as the Cr concentration increases above 10 at.%. Thus, the ordering tendency of Fe and Cr is inversed as the concentration is increased. It is worth to mention that an inversion of the ordering behavior in this material has been observed previously at much higher temperatures by means of diffuse-neutron-scattering and resistivity measurements [10, 11] as well as Mö ssbauer studies after electron irradiation [12]. In these works, α_i was found to change sign from negative to positive at a Cr concentration of 10 at.%. It is here shown for the first time that this ordering behavior is present at much lower temperatures when the alloy is irradiated. This effect is of critical importance for the stability of the material under irradiation, since the enhanced atomic diffusion in conjunction with the ordering tendency may accelerate atomic processes that lead to the degradation of key macroscopic physical and mechanical properties like strength, toughness and thermal response.

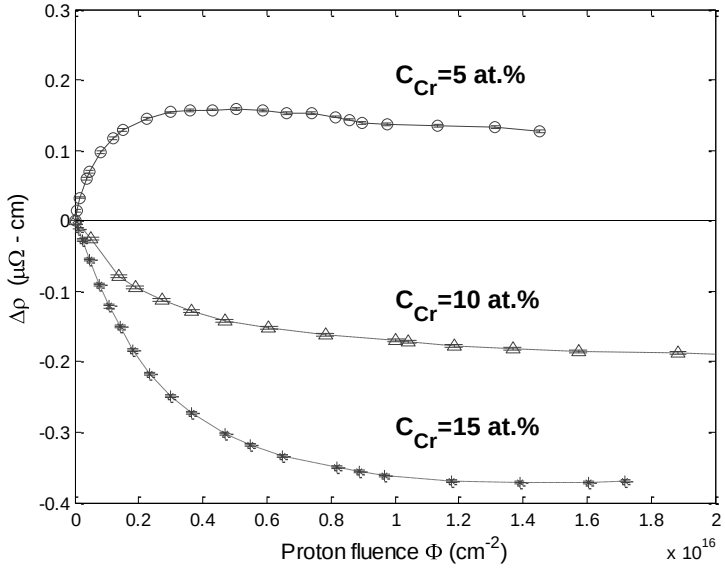

Fig. 3. Change of the electrical resistivity of $Fe_{1-c}Cr_c$, with c=5, 10, 15 at. % Cr concentration, as a function of fluence during 5MeV proton irradiation at high temperature.

4. Conclusions

Changes in the electrical resistivity of pure Fe and Fe-Cr alloys during irradiation by energetic protons have been measured at 40 and 400 K. At low temperature the resistivity of Fe and Fe-Cr increases linearly with irradiation dose, due to the accumulation of radiation damage. At 400 K this damage is instantly recovered in pure Fe by fast defect migration and annihilation. In Fe-Cr alloys the irradiation at 400 K causes a redistribution of Cr solute atoms which leads to a transient behavior of the resistivity. For low Cr concentration the resistivity transient is positive, while for Cr concentration higher than 10 at.% it becomes negative. This is correlated to the ordering tendency of Cr in Fe which is also known to change sign at about 10 at.%. Our experiments show that in Fe-Cr alloys under irradiation changes in the ordering of Cr solute atoms is occuring even at relatively low temperatures. Further studies as a function of irradiation temperature and dose are needed to fully understand this important effect.

Acknowledgments

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