Ion implantation induced defects in Fe-Cr alloys studied by conventional positron annihilation lifetime spectroscopy

This content has been downloaded from IOPscience. Please scroll down to see the full text.

2011 J. Phys.: Conf. Ser. 265 012014
(http://iopscience.iop.org/1742-6596/265/1/012014)

View the table of contents for this issue, or go to the journal homepage for more

Download details:

IP Address: 37.16.72.140
This content was downloaded on 20/07/2015 at 21:27

Please note that terms and conditions apply.
Ion implantation induced defects in Fe-Cr alloys studied by conventional positron annihilation lifetime spectroscopy

V Kršjak¹, S Sojak², V Slugeň² and M Petriska²

¹ Joint Research Centre, Institute for Energy, European Commission, P.O. Box 2, 1755 ZG Petten, The Netherlands
² Department of Nuclear Physics and Technology, FEI, Slovak University of Technology, Ilkovičova 3, 812 19 Bratislava, Slovak Republic

E-mail: vladimir.kršjak@ec.europa.eu

Abstract. The influence of chromium on the radiation damage resistance of the iron based alloys has been studied using conventional positron annihilation lifetime spectroscopy (PALS). Experimental data evaluation has been supported by the former theoretical calculation of positron lifetimes in the studied materials and well-defined types of defects. For this purpose, density functional theory (DFT) computation method has been applied. The spectrum of used ²²Na positron source was decomposed into discrete fractions to better calculate efficiency of near surface layers study. For the experimental simulation of α-radiation and obtaining of defined cascade collisions in the materials, helium implantation was used. Different level of the implanted dose (6.24 × 10¹⁷ – 3.12 × 10¹⁸ cm⁻²) corresponds to local damage up to 90 DPA acquired in thin <1 µm region. Experimental measurement has been performed using the PALS technique on the four different Fe-Cr binary alloys (2.36; 4.62; 8.39; 11.62 wt% of Cr). The results showed that chromium has a significant effect on the size and density of the implanted defects and specific Cr content should prevent the vacancy clusters formation.

1. Introduction
Conventional positron lifetime technique based on ²²Na positron source is usually applied for study of the bulk of the specimen. Regular surface (or near surface) study requires slow positron beam with adjustable energy to observe specific depth of the specimen. Nevertheless, considerable fraction of positrons is absorbed in the near surface region, where the implantation profile decreases very fast [1]. This paper discusses our recent positron annihilation lifetime experiments focused on the radiation treated Fe-Cr alloys. To simulate high neutron fluencies in these materials, the helium implantation has been used. The interpretation of the measured data has been based on previous theoretical calculation of Troev et al. on pure iron and chromium [2, 3].

Fe-Cr binary alloys have been chosen for our study of chromium influence on the radiation damage resistance. This alloying element is an important component of the reactor structural materials since the 1970’s. However only high chromium ferritic/martensitic steels application in the nuclear industry (fast reactors) showed the importance of this element on the material properties after radiation treatment [4]. In particular the decrease of the void swelling due to chromium addition has been often discussed in the last decade [5, 6]. Although this role of chromium in swelling resistance is already quite known, the complex impact on the radiation damage resistance is not fully understood yet.
2. Experimental

2.1. Materials and sample preparation
To study the influence of chromium concentration, four Fe-Cr binary alloys with different Cr content have been selected. The detailed chemical composition of the alloys can be seen in table 1. All the alloys were heat-treated to ensure well defined martensitisation. More detailed information about these materials and the fabrication processes can be found in [7].

| Table 1. Chemical composition of the studied materials. |
|----------------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|
| Alloy ID | Cr* | O* | N* | C* | Mn | P | Si | Al | Ti | Ni | Cu | V |
| L251 | 2.36 | 0.035 | 0.012 | 0.008 | 0.009 | 0.013 | 0.002 | 0.003 | 0.004 | 0.044 | 0.005 | 0.001 |
| L259 | 4.62 | 0.066 | 0.013 | 0.02 | 0.02 | 0.011 | 0.006 | 0.003 | 0.003 | 0.06 | 0.01 | 0.001 |
| L252 | 8.39 | 0.067 | 0.015 | 0.021 | 0.03 | 0.012 | 7E-04 | 0.007 | 0.003 | 0.07 | 0.01 | 0.002 |
| L253 | 11.62 | 0.031 | 0.024 | 0.028 | 0.03 | 0.05 | 0.006 | 0.003 | 0.004 | 0.09 | 0.01 | 0.002 |

* measured after heat treatment

The as-received materials have been cut to the desired dimensions, grinded and carefully polished to mirror like surfaces (polishing paste – grade 1 μm) before exposure to helium implantation. Subsequently, an untreated reference sample and the implanted samples were investigated with PALS techniques.

2.2. Radiation treatment
To obtain cascade collisions in the microstructure of the studied materials without neutron activation, accelerated helium ions have been used. Implantations of 250 keV helium ions at five different fluence levels have been performed at the linear accelerator of the Slovak University of Technology in Bratislava [8]. The depth profile of collision events can be seen in figure 1. The DPA (displacement per atom) parameter has been calculated for the volume of 99% positron absorption (see table 2).

| Table 2. Calculations of average DPA for different level of implantation in first 100 μm layer (DPA_{PALS}) of studied Fe-Cr alloys. |
|----------------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|
| Dose [ions/cm²] | 6.24×10¹⁷ | 1.25×10¹⁸ | 1.87×10¹⁸ | 2.5×10¹⁸ | 3.12×10¹⁸ |
| [C/cm²] | 0.1 | 0.2 | 0.3 | 0.4 | 0.5 |
| DPA (99% positron absorption) [a.u.] | 0.15 | 0.30 | 0.45 | 0.60 | 0.74 |
| DPA (Bragg peak) [a.u.] | 18.55 | 37.10 | 55.64 | 74.19 | 92.74 |

Figure 1. Depth profile of the helium implantation, \( E = 250 \) keV (SRIM simulation of \( 10^6 \) ions).

2.3. Application of conventional PALS technique
Positron lifetime measurements have been performed on the fast-fast coincidence setup of experimental equipment. Typical measurements took six hours and the time resolution of the apparatus
was determined to be less than 210ps. According the long term measurements of iron based alloys, uncertainty of the results $\tau_2$, $I_2$ and MLT were estimated to be ±5ps, ± 10% and ±3ps. The experimental data have been evaluated with the LT ver. 9.0 program [9] using the three component decomposition. This spectra fitting was sufficient (fit’s variance < 1.1 in almost all cases) and moreover the fourth component cannot be found in any spectra. However this approach means that only one component describes the lattice defects and other two components correspond to material bulk and the long component with small intensity (< 1.5%) is typical for the conventional PALS source-sample setup. Therefore the $\tau_2$ component may differ from known positron lifetimes of particular lattice defects and combination of several types of defects must be considered by this data interpretation.

3. Results
The PALS results show, that positron lifetime in the defects which correspond to the size of the vacancy type defects (vacancy clusters) is increasing with the implanted dose in all specimens (figure 2a). For the low Cr alloys (L251, L259) there is a clearly defined maximum for the fluency about 1.87×10¹⁸ ions. The highest positron lifetime (the largest clusters of vacancies) was measured for 2.56% of Cr alloy (L251) and its value 235ps should correspond to a cluster of 4 – 5 empty vacancies or slightly larger clusters containing helium. However, the absolute value of this parameter may be underestimated due to fact that here are included initial defects, typical for tempered ferritic/martensitic steels [10] and only certain number of positrons annihilated in the ion implantation damaged layer. Higher Cr content alloys (8.39 and 11.62% of Cr) are more resistant to creating of such a large vacancy clusters which can be seen via lower positron lifetime $\tau_2$. From this aspect, Fe8.39%Cr specimen has the most optimal chemical composition for the radiation damage resistance and correspondent also with the choice of currently most perspective materials for advanced nuclear power systems (e.g. EUROFER97 – 8.96% of Cr). The intensity of the annihilation in defects (figure 2b) shows characteristic behavior for the Fe8.39%Cr as well. The significant intensity peak for this alloy can be interpreted as a higher density of the uniformly distributed small defects. This state is more acceptable from the structural point of view than the clustering of these defects into large voids, which can be seen from figure 2a for low-Cr alloys and which may lead to radiation embrittlement.

Figure 2. Positron annihilation in defects. Positron lifetime $\tau_2$ (a) and intensity $I_2$ of annihilation (b).

The MLT parameter is increasing with the implantation dose in all materials (figure 3a). It confirms the fact that conventional PALS technique has been successfully applied on the near surface region study. Different slopes of the MLT curves indicate unequal behavior of the materials under radiation treatment. The plane projection of the MLT dependency (figure 3b) shows that Fe-Cr alloys with Cr content 5 – 7 wt% seems to be most resistant to the ion implantation induced defects.
Figure 3. Positron mean lifetime (MLT) in the He implanted Fe-Cr alloys. 3D view (a) and its planar projection (b).

4. Conclusions
Our study showed that conventional positron lifetime technique can be used also with advantage for study of near surface layers damaged by light ion implantation.

Performed measurements showed that chromium plays an important role in the formation of the microstructure under radiation treatment. In particular, chromium content in Fe-Cr alloys has an influence on the vacancy cluster forming. In the low chromium alloys the small vacancy clusters have been observed. However, such clusters of the vacancies have not been observed in high chromium materials as long as presented DPA values are considered. From the studied materials, the Cr concentration on the level about 9 wt% was evaluated as a most suitable having in mind the resistance to the radiation induced defects agglomerations. Mono- and/or di-vacancies are more acceptable than their agglomerations from the radiation embrittlement resistance point of view.

Acknowledgement
This project was supported by VEGA 1/3188/06 and 7RP-Euratom/CU.

References
[1] Dryzek J and Sieracki J 2007 Nucl. Instr. Meth. B 258 493
[2] Troev T, Markovski A, Peneva S and Yoshiie T 2006 J. Nucl. Mater. 359 93
[3] Petrov L, Nankov N, Popov E and Troev T 2008 AIP Conf. Proc. 996 177
[4] Klueh R L 2004 Elevated-temperature Ferritic and Martensitic Steels and their Application to Future Nuclear Reactors, ORNL/TM-2004/176 report
[5] Porollo S I, Dvoriashin A M, Vorobyev A N and Konobeev Yu V 1998 J. Nucl. Mater. 256 247
[6] Konobeev Yu V, Dvoriashin A M, Porollo S I and Garner F A 2006 J. Nucl. Mater. 355 124
[7] Matijasevic M and Almazouzi A 2005 Modellisation of Irradiation Effects: Modeling Oriented Experiments on Fe-Cr Model Alloys, SCK•CEN-R- 4196 report
[8] Kovác P, Pavlovic M and Dobrovodský J 1994 Nucl. Instr. Meth. B 85 749
[9] Kansy J 1996 Nucl. Instr. Meth. A 374 235
[10] Matijasevic M and Almazouzi A 2008 J. Nucl. Mater. 377 147