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Advanced positron annihilation studies of CuCrZr alloys for fusion technology

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Advanced positron annihilation studies of CuCrZr alloys for fusion technology

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1 Introduction
The precipitation as a consequence of applied heat treatment (HT), observed by Transmission Electron Microscopy (TEM) [1], was the motivation to perform the study of the CuCrZr structure using Positron Annihilation Spectroscopy (PAS). The reasons, why to do that, were at first to observe the HT effect by a non-destructive evaluation method, secondly to see the annihilation sites of positrons in non-irradiated CuCrZr material and at last but not least to perform PAS measurements in order to have reference values for non-irradiated material as a starting point for subsequent investigations of neutron irradiated CuCrZr alloys.
Positron annihilation spectroscopy (PAS), as non-destructive evaluation method, is sensitive to trapping sites such as open volume defects (vacancy, vacancies agglomerates, Stacking Fault Tetrahedra SFT, dislocations, grain boundaries GB, voids), where the electron density is less than in the bulk [2]. We concentrate on the results obtained by PAS on non-irradiated CuCrZr samples with the aim to investigate the HT effect. We have therefore measured and evaluated the PAS lifetime spectra of the heat treated CuCrZr samples using the same heat treatment conditions.

2 Experiment design
The composition of the studied material (supplied by Outokumpu Oyj, Finland) is Cu-0.78%Cr, 0.13% Zr, 0.003% Si, 0.008% Fe. The contents of Cr and Zr were changed in the course of fusion materials research for ITER first wall because of their influence on the precipitation. Studies of CuCrZr are world-wide still in progress [3].
The procedure of heat treatment is summarized in Table 1. The specimens were heat treated in an oven at a vacuum at the level of < 1.33 mPa (10-5 torr) and then water quenched.

Table 1 –Summary of the heat treatments for CuCrZr (Outokumpu)
Heat Treatment
(HT) Description of applied HT

SA Solution annealing (SA) at 960⊠C for 3 h followed by water quench (WQ)
PA Prime ageing (PA): SA + ageing at 460⊠C for 3 h followed by WQ
PA+HT1 PA + ageing at 600⊠C for 1 h + WQ
PA+HT2 PA + ageing at 600⊠C for 4 h + WQ
PA+HT3 PA+ ageing at 700⊠C for 4 h + WQ
PA+HT4 PA+ ageing at 850⊠C for 4 h + WQ
PA+HT5 PA+HT4+960⊠C for 3 h + WQ

3 Transmission Electron Microscopy
The main results, quantitatively listed in Table 2, are shown for comparison with the present study of the heat treatment effect in non-irradiated CuCrZr alloy.
Table 2 –Summary of the TEM results observed for CuCrZr (Outokumpu).
Heat Treatment
(HT) Results from TEM
Density of Precipitates
[m-3] Average Size of Precipitates
[nm]
SA - -
PA 2.6 ⊠ 1023 2.2
PA+HT1 1.7 ⊠ 1022 8.7
PA+HT2 1.5 ⊠ 1021 21.3
PA+HT3 7.0 ⊠ 1020 46.4
PA+HT4 - -
PA+HT5 - -

The heat treatments (HT1, HT2 and HT3) after prime ageing (PA) led to significant coarsening of the prime aged precipitate microstructure. The overageing (HT4) at 850°C for 4 hours caused almost complete resolution of the precipitates, it led to very low density of rather large and heterogeneously distributed precipitates and therefore no quantitative measurements were made.

4 Positron Annihilation Spectroscopy
A conventional two-detector positron lifetime system was used for the measurements. The positron source Na-22 in nickel foil (activated NaCl solution dropped on, dried and encapsulated in 0.5 mg/cm2 thick Ni foils) was placed between two samples.
In total, there were performed 69 (46 at CuCrZr and 23 at reference Cu sample) positron lifetime measurements, with more than 3.5×106 events in each measured spectrum. The measurements of the 7 differently heat treated CuCrZr samples, listed in Table 1, were performed several times for each sample set. The time resolution curve (TRC) of the spectrometer was well defined by 2 Gaussians (FWHM ~ 234 ps).
Positrons are sensitive to sites with less electron density such as defects and may become trapped by them. The trapping model provides simple view onto the effect of positron trapping by defects in the metal structure. In its simplest version the model assumes that positrons initially are in the bulk of the material where they annihilate with the rate λb= τb-1. From the bulk they may become trapped in one type of defects with a time independent trapping rate κ. For a trapped positron the annihilation rate is λd= τd-1. These assumptions lead to the following rate equations for the disappearance of positrons in the bulk and in the trap [2]:
The measured PAS lifetime spectra could be decomposed into two lifetime components, τ1 and τ2 with corresponding intensities I1 and I2 (I1+I2=100%), shown in Figures 1 and 2, respectively. The values obtained for CuCrZr alloy are compared with the bulk value of positron lifetime in pure copper Cu (110 ps). By application of simple trapping model with bulk value τb=110ps the values of τ1calc (Fig. 1 and Fig. 2) and of trapping rate κ (Fig. 3) into the trap (defect) were calculated. The dependency of the specific trapping rate on the precipitates radius rppt was then estimated (Fig. 4).

Fig. 1 –The HT effect investigated by PAS measurements for non-irradiated CuCrZr alloy samples (Table 1). The positron lifetime components: τ1 –shortest lifetime, τ2 –long defect lifetime, τ1calc –calculated τ1 value derived from the trapping model.

Fig. 2 –The intensity I2 of the long-lived component (due to positrons trapped in defetcs) in the PAS spectra for non-irradiated CuCrZr (Outokumpu) alloy for various heat treatments (Table 1).

Fig. 3 –The trapping rate ⊠ with which positrons are trapped into defects in the non-irradiated CuCrZr (Outokumpu) alloy.

Fig. 4 –The experimental values of the specific trapping rate ⊠ppt (diamond line), deduced from the simple trapping model.

5 Conclusions
In present work, the comparison of results observed by TEM and PAS techniques at CuCrZr (Outokumpu) alloy was performed. In the PAS measurements a well-defined long defect lifetime τ2 was observed with basically constant value over the whole range of HT (176⊠8 ps), except the SA and PA+HT5 states, where τ2 is slightly higher (200⊠15 ps), while its intensity I2 exhibited strong dependency on the HT. Thus, a clear influence of the applied HT on the defect microstructure of CuCrZr alloy was observed. The measured defect lifetime τ2 is associated with the creation of the vacancy-type defects at the precipitate matrix interface, believed to arise from lattice mismatch between matrix and precipitates. The dependency of positron trapping rate on the precipitates radius rppt was estimated. The experimental values, measured at the non-irradiated CuCrZr alloy, represent the starting points for the following measurements at neutron irradiated CuCrZr samples.

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