DEFECT TYPES IN NOBLE METALS MIGRATING IN STAGE III  
AFTER IRRADIATION AND QUENCHING

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By combination of quenching and irradiation with different particles, we have determined the nature of trapped defects at $^{111}$In in Cu and Au, observed by the perturbed angular correlation technique. The defects were identified by changing the damaging conditions and measuring all parameters of the quadrupole interaction: electric field gradient (efg), asymmetry parameter, orientation of the efg, and temperature dependence of the efg. Preliminary experiments on $^{111}$In doped Ag have been performed.

Microscopic methods, such as perturbed angular correlation (PAC) or the Mössbauer effect offer the possibility of distinguishing between different types of defects migrating within the same temperature range. Unfortunately, it is not possible to identify a trapped defect by comparing the measured electric field gradient (efg) with that calculated via a proper theory, but the possibility of recognizing the same defect under different experimental conditions allows one to determine the nature of the defect. This is shown in the following for defects trapped in Cu, Au and, to a limited extent, in Ag.

First, we want to report the attributes of a trapped defect which are measurable via PAC:

- the component of the efg $e_{zz}$ via the modulation frequency of the measured time spectrum:
  \[ \nu_Q = \frac{e^2 Q_{zz}}{n}, \]  
  (\(eQ\) = Quadrupole moment, \(h\) = Planck's constant)

- the asymmetry parameter \(\eta\), which describes the deviation from the axial symmetry of the efg:
  \[ \eta = \frac{e_{xx} - e_{yy}}{e_{zz}} \]  

- the fraction \(f\) of radioactive probe atoms with trapped defects via the amplitude of the modulation frequency of the time spectrum,

- the orientation of $e_{zz}$ with respect to the lattice directions,

- the temperature dependence of the efg.
Information about the nature of trapped defects can be extracted after different damage-inducing processes:
- irradiation with electrons should especially favour the formation of simple defects, e.g. single vacancies and interstitials,
- quenching demonstrates the vacancy character of trapped defects,
- changing the concentration of trapping centers in the sample (via the concentration of probe atoms) or the concentration of defects (via the irradiation dose) changes the formation probability of different defect types at the probe atoms in a different manner.

In the following, we will use all this information to clarify the defect situation in stage III of Cu, Au and Ag.

The samples (Au: COMINCO 99.9999%, Cu: ASARCO 99.9998%, Ag: MRC 99.999%) were doped with $^{111}$In by implantation at 350 keV or diffusion under $H_2$-atmosphere. The implanted samples were annealed at about 780 K to remove the damage introduced by the implantation. Afterwards, the doped samples were irradiated with protons or heavy ions at our 350 keV implantation facility at 80 K. The electron irradiations (3 MeV) were performed at the Van-de-Graaff accelerator of the Kernforschungsanlage Jülich. The quenching experiments were carried out in an apparatus as described in [1]. All samples were measured at 77 K in an isochronal annealing sequence with 10 min heating time for each annealing step.

The PAC measurements were performed with a standard 4-detector setup, and the relevant parameters ($\nu_Q$, $n$, orientation, $f$, T-dependence) were extracted from the recorded time spectra in a way described earlier [2]. The orientation measurements were performed at Cu and Au single crystals (MRC) prepared by implantation with $^{111}$In in the same way as the polycrystalline samples.

Copper: In an earlier work [3], we showed that two vacancy-like defect configurations are formed at $^{111}$In in stage III, which are characterized by $\nu_Q = 116$ MHz, $n = 0$ and $\nu_Q = 181$ MHz, $n = 0$. We interpreted them as a trapped monovacancy and a trapped small vacancy cluster. We performed additional experiments on the structure of these defects. For this purpose, two different types of experiments were performed. The first consists of an electron irradiation with different doses and different In concentrations. Fig.1 shows the result of this experiment: Two samples with an $^{111}$In concentration <10$^{-2}$ ppm were irradiated at 4.2 K to Frenkel pair concentrations equivalent to a resistivity increase of 16 $\mu\Omega$cm and 113 $\mu\Omega$cm. In the low dose case (upper diagram), the defect 1 is predominantly formed, whereas defect 2 is nearly invisible; in the second case (middle) defect 2 reaches the same fraction as defect 1. This shows that defect 1 should be the simpler one of the two defects. (Both experiments are part of Ref. [3]). A third Cu sample was also irradiated with a high dose (120 $\mu\Omega$cm) (lower diagram in Fig.1), but in this case the sample was additionally alloyed with 20 ppm inactive In. The result of this experiment is a reduction of defect 2 and an increase of defect 1. This behaviour shows that defect 2, the small vacancy cluster, is formed via the simple defect 1, the monovacancy, by multiple trapping of vacancies, probably a second vacancy, so that $\nu_Q$ describes a trapped divacancy. As a further result, not observed in the other two samples, we find a "crossover" of the two fractions around 230 K. This behaviour confirms what we stated in Ref. [3], that defect 2, the divacancy, already exists in the lattice and is more mobile than the monovacancy. That means that up to 230 K $\nu_Q$ is caused by the trapping of mobile divacancies and above 230 K $\nu_Q$ is formed via the monovacancy ($\nu_{01}$) by trapping of a second vacancy. - To get more information concerning the trapped monovacancy we performed a second type of experiment, the measurement of the efg-orientation belonging to this defect. The experimentally determined $e_{dzz}$-component of the efg tensor points in <110> direction as expected for a monovacancy trapped in the nearest neighbourhood of $^{111}$In.