Temperature-dependent positron trapping in copper and aluminum tubes after tensile deformation

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Doppler-broadening measurements on aluminum and copper polycrystals were performed *in situ* during tensile tests and temperature-dependent after different degrees of deformation. The sensitivity limit of positrons to defects produced by plastic deformation was found at a resolved shear stress of $\tau = (4 \pm 1)$ MPa in aluminum and $\tau = (9 \pm 1)$ MPa in copper. This corresponds to dislocation densities of $\varrho \approx 2 - 3 \times 10^{12}$ m^{-2}. Temperature-dependent Doppler-broadening measurements revealed distinct differences of the positron trapping at dislocations in copper after weak and strong plastic deformation. The transition of the trapping behavior occurred between $\tau = 20$ and 30 MPa. In aluminum such a dependence of the positron trapping behavior on the degree of deformation was not observed. The measurements were analyzed in terms of a three-state trapping model which provided numerical values for the trapping rates and the binding energy of the positron to dislocation lines. [S0163-1829(99)03625-5]

I. INTRODUCTION

In order to understand the capability of metals to undergo heavy plastic deformation a large variety of methods has been developed to study their microstructure and to investigate the underlying microscopic processes. Some of those methods, like transmission electron microscopy, have been applied very succesfully since dislocations as the vehicles of plastic deformation and other defects in the crystal lattice can be directly imaged. Other methods, for example, magnetic methods are rather indirect and not generally applicable, however, they allow a nondestructive examination of specimens.[1] For engineering applications, however, just nondestructive methods are of crucial importance for inspection of plant components, for microstructure characterization and residual lifetime assessment. A recent review of these methods with their strong and weak points has been given by Dobmann *et al.*[2] There is still a lack of nondestructive techniques which are applicable to plant components and are able to detect the early stages of the formation of ''microporosity.'' Physically speaking this is the formation of vacancy agglomerates especially along grain boundaries to be brought about by dislocation processes. Thus, positron annihilation, which is known to be sensitive to dislocations and vacancies, would be a potential method to increase the detection sensitivity in nondestructive testing, and it is, in principle, also capable of field application.[3] Furthermore, positron-lifetime measurements provide the possibility to get defect specific information, if the data can reliably be separated into a spectrum of several defect specific positron lifetimes. Moreover, it is not sensitive to residual stresses leading to local variations of the lattice constant which is the dominating effect in examinations by x-ray defraction and neutron scattering.

The sensitivity of positrons to defects in plastically deformed metals has been well-known since the 1960s.[4,5] A summary of the first two decades of research has been given by Byrne.[6] The interaction of positrons with different types of crystal defects has been subject of intense research.[7–10] Positron capture in single vacancies is probably the best understood process because vacancies can be created and investigated in thermal equilibrium at elevated temperatures with negligible disturbances by other defect types. Plastic deformation does not exhibit a similar advantage because the production of dislocations and vacancies is always interconnected,[11,12] which challenges experimental investigations. Depending on the mobility of vacancies they may be eliminated by moving to sinks, such as surfaces, grain boundaries, and dislocations or they may form clusters. But even their successful elimination may create new problems for positron investigations on dislocations since it is difficult to check if the atomic configuration of dislocation lines has changed by the formation or elimination of monoatomic jogs through the absorption of vacancies and interstitials. Therefore, it is necessary to start with very weak degrees of deformation ($\varepsilon < 0.01$) in order to produce mainly dislocations and to keep the accompanying vacancy concentration to a minimum. Ideally, the dislocations should be homogeneously distributed. The best experimental approximation of this situation is achieved by weak, uniaxial tensile deformation before dislocation bundles become too dense or cell structures start to form.[13–15]

In spite of all difficulties, an improved knowledge of the positron dislocation interaction is essentially required for any interpretation of positron annihilation measurements on plastically deformed materials.

The state of the art has recently been summarized by the present authors.[16] Experiment and theory agree that dislocations are shallow positron traps with binding energies of $E_b \lesssim 100$ meV.[17–20] The positron lifetime associated with dislocations is just slightly smaller than that of single vacancies.[21–23] This has led to the idea to consider dislocation lines acting as precursor states for the transition into deeper traps such as jogs and vacancies bound in the stress field around dislocations.[17,24,25] The trapping rate into these

associated defects which are often described as *vacancy like defects* is enhanced by the presence of dislocations.[21,26,27] The consequences of this model, especially on the temperature dependence of the positron trapping rate at dislocations, were discussed by several authors.[19,28,29] Trapping at dislocations appears either by enhanced trapping in associated defects or by reduced trapping in defects not associated with dislocation lines. The present data were analyzed in terms of this general model and both situations were observed depending on the degree of deformation.

The present paper deals with Doppler-broadening measurements of positron annihilation in aluminum and copper polycrystals performed (i) *in situ*, during tensile testing at room temperature, and (ii) temperature dependent between 13 and 263 K in different stages of deformation. Additionally, Doppler-broadening measurements were performed after annealing treatments.

Thanks to the *in situ* technique, the response of the Doppler broadening on the evolution of the defect structure during tensile deformation could be observed on a single specimen. The classic so-called ''sandwich'' method requires two flat pieces of the same specimen between which a positron source is sandwiched. This would compel us to establish different stages of microstructure evolution in different tensile specimens since we would have to slice, i.e., to destroy them in order to get the two pieces required. Alternatively, one could establish subsequent deformation stages in two small platelets to be used for the ''sandwich'' by gradual cold rolling, which has, however, the disadvantage of forming complicated dislocation arrangements. If one wants to profit from the comparatively simple and clear microstructure established in a tensile test, one has to reduce the scatter owing to slight differences in the initial dislocation content or effects related to the polycrystalline structure. Further, cylindrical specimens should be prefered to flat ones since their deformation behavior is not susceptible to edge effects. Although tensile tests are in general well reproducible, such slight differences give rise to slightly different hardening curves, especially at the beginning of the tensile test, when the specimens are still soft. Since positrons are very sensitive to defects produced by plastic deformation this specimen variability will be reflected in a scatter of the positron-annihilation measurements. It is the main advantage of the nondestructive *in situ* technique that all stages of microstructue evolution are part of the same deformation history. Thus, scatter due to specimen variability can be avoided. The method applied in this paper improves the comparability and reliability of the results and is economic. The *in situ* measurements reveal the sensitivity of the positron-annihilation technique to defects produced by plastic deformation and allow the proper selection of the different deformation stages to be examined at variable temperature in order to study positron trapping in shallow traps.

After a short presentation of the experimental methods in Sec. II, the results of the measurements are presented in Sec. III. An interpretation of the results is given in Sec. IV on the basis of a three-stage trapping model. The discussion in Sec. V demonstrates that such a model can provide a satisfactory description under some reasonable assumptions.
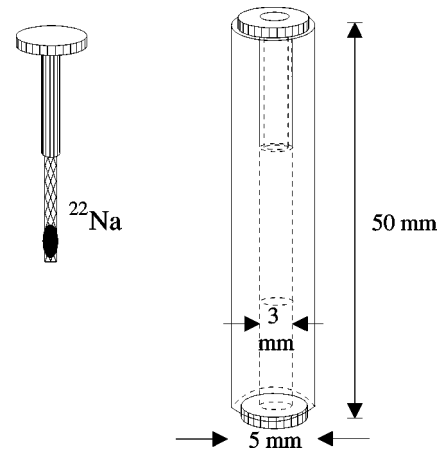

FIG. 1. ^{22}NaCl deposited on and sealed by a 2 μm thin Mylar foil was used as positron source. The source was attached to one of the stainless-steel inserts which were used to close the tubular-shaped specimens in order to avoid squeezing them when clamped into the grips of the tensile test machine. Thus, during the measurements the source was inside the specimen.

II. EXPERIMENTS

A. Specimen preparation

Tubular-shaped specimens of 5 mm outer and 3 mm inner diameter and 50 mm length were produced from polycrystalline, highly pure (99.999%) copper and aluminum rods purchased from Alpha Chemicals/Johnson Matthey Ltd. In order to eliminate crystal defects, the copper and aluminum tubes were annealed under vacuum ($p \leq 2 \times 10^{-5}$ hPa) for 4 h and then slowly cooled to room temperature within a period of 6 h. The annealing temperatures were 1200 and 870 K for the copper and the aluminum tubes, respectively. After these annealing treatments large grains of about 1 mm in dimension were obtained. Therefore, the contribution of positron trapping in grain boundaries can be neglected.[30]

Tubular-shaped specimens were chosen because they allow to enclose the positron source during the tensile tests. Thus, nondestructive measurements of the Doppler broadening of the 511 keV annihilation line were possible *in situ*, during deformation. For this purpose the positron source was fixed to one of the two stainless steel inserts (see Fig. 1). These were fitted into each tube to preserve its shape when the specimen was mounted into the piezoelectric deformation machine which has already been described in detail elsewhere.[16] During the temperature-dependent positron-annihilation measurements a direct immersion of the source to the helium flow and a contamination risk of the cryostat was avoided. Moreover, this method allowed to pull out the source of the deformed tube and to subject the specimen to an annealing treatment in order to gain additional information on the defects.

B. Plastic deformation

Tensile tests were performed with a piezoelectric deformation machine which enabled precisely controlled deformation at very small plastic strains and strain increments.[16] The mechanical data, engineering strain $\varepsilon = \Delta l / l$ and the applied stress $\sigma = F/A$ were recorded. Herein, Δl denotes the length

increase along the freely deformable length l of the tube between the grips which was 18 mm. F denotes the force acting along the axis of the tube and A the cross-sectional area filled by the tube material.[31] The tests were performed with a strain rate $\dot{\varepsilon} = 5 \times 10^{-5}$ s^{-1}.

In order to allow us to compare the results obtained on the polycrystalline tubes with those obtained earlier on single crystals[16] the axial flow stress σ was converted into the shear flow stress τ resolved to the primary glide plane of the dislocations using the relation

$$\tau = \frac{\sigma}{M}, \tag{1}$$

where $M = 3.06$ denotes the so-called Taylor factor. The Taylor factor demands that individual grains deform by multiple slip in order to accomplish compatibility.[32] According to continuum mechanical considerations by Kröner[33] the transition from single to multiple slip may be expected in the plastic deformation range $10^{-4} \leqslant \varepsilon_{pl} \leqslant 10^{-3}$. Thus, the Taylor factor should provide a reasonable comparison between single crystal and polycrystal data in the whole range of interest. A quantitative comparison is, however, only required when the dislocation density at the sensitivity threshold is estimated from the corresponding resolved shear stress values.

C. Doppler-broadening measurements

The positron source had an activity of 2 μCi ^{22}NaCl and was deposited on a 2 μm thin Mylar foil which was attached to one of the stainless steel inserts (Fig. 1). The γ-ray spectra were recorded by a Ge detector with an energy resolution of 1.24 keV at 514 keV and comprised about 1.7 $\times 10^6$ events in the 511 keV annihilation peak.

The shape of the 511 keV annihilation line is quantitatively described by the S parameter which is defined as the ratio of two integrals, $S = A/B$. Herein, the integral A expresses the number of counts in a symmetrically located central region of the annihilation line and B is the integral number of counts in the whole line. The boundaries of the integral A are chosen so that $S \approx 0.5$ in the well-annealed specimen.

The physical relevance of the S parameter is based on the momentum difference between valence and core electrons. The core electrons localized in the ion core have a much higher momentum than the valence electrons. Owing to the locally reduced density of core electrons in vacancylike defects such as vacancies, vacancy clusters or lattice dilatations, e.g., around ''text-book'' edge dislocation lines, positron traps exhibit a lower density of electrons with high momentum. Thus, the probability of positron annihilation with a low-momentum valence electron is increased compared to the situation in the perfect lattice. This causes an increased number of counts in A, i.e., with small Doppler shifts of the annihilation energy. Consequentely, the S parameter is larger in a specimen containing vacancylike defects than in a defect-free material.

The temperature dependence of the S parameter denoted by $S(T)$ yields further information on the presence of shallow traps which may trap positrons at low temperarture, whereas thermal detrapping may occur at higher temperature.

In situ Doppler-broadening measurements were carried out in the loaded state and at strain rate zero ($\dot{\varepsilon} = 0$) in order to avoid microstructure changes during recording of the γ-spectrum. Stopping tensile straining is related with a stress relaxation of a few MPa, which is completely recovered when straining is subsequently continued. At selected stress levels the effect of elastic deformation on the positron annihilation characteristics was investigated by comparing the Doppler-broadening measurements in the loaded state to measurements after complete removal of applied stress. A significant difference has not been observed.

The *in situ* technique allowed a series of Doppler-broadening measurements separated by small deformation increments. The specimen could remain in the deformation machine and no frequent dismounting-mounting cycles were required. In this way the risk of unintentional deformation during handling of the specimen could be avoided which is especially high at the beginning of the tensile test when the specimen is not yet hardened. Thus, we were able to follow the development of the Doppler broadening in the same specimen during tensile testing and to select the interesting deformation stages for measurements at variable temperature in a cryostat. For this purpose the specimen was removed from the deformation machine in several stages of the deformation and placed in a helium flow cryostat where the temperature dependence of the Doppler broadening was examined between 13 and 263 K in steps of 10 K.[34]

In order to obtain further information on defects, additionally, annealing treatments of weakly and strongly deformed specimens were carried out at various annealing temperatures and were combined with temperature-dependent Doppler-broadening measurements in the cryostat.

III. RESULTS

The interpretation of our results takes into account that positrons experience dislocation lines as objects which offer two different types of traps. First, let us say that the true dislocation line in the sense of plasticity theory is a shallow trap and becomes efficient only at low temperatures when thermal detrapping is suppressed. It is characterized by S_D. Second, deep traps like jogs on dislocation lines, and vacancies trapped in the stress field around dislocation lines cannot be distinguished from vacancies in the lattice. Those vacancies associated with dislocations, however, are supposed to have a higher trapping rate due to the dislocations acting as precursor states for a transition into deeper traps. The deep, vacanylike traps are characterized by S_v. Thus, we have to deal with positrons annihilating in the perfect lattice, characterized by S_b, along true dislocation lines and in deep traps. If we consider that the annihilation rates obtained for true dislocations λ_D and for the bulk λ_b are approximately identical,[19,20] we may tentatively assume that positrons experience a similar electronic environment in a true dislocation line and in the perfect lattice. We may further assume that thermal expansion of a crystal affects the bulk and the environment of a dislocation in the same way. Thus, we may expect also the same temperature dependence for the S parameter in the bulk and along true dislocation lines, i.e., $S_b(T) = S_D(T)$. With these ingredients of our trapping model, which is outlined in detail in Sec. IV, we can restrict our
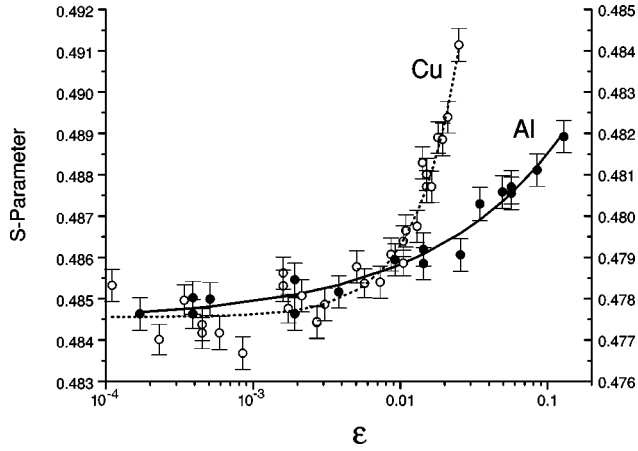
FIG. 2. The S parameter (Ref. 37) as a function of the strain ε (logarithmic scale) for copper (Cu-1) and aluminium polycrystals; right and left axis, respectively. The curves correspond to least-squares fits to the power law $S(\varepsilon)=S_b+\alpha\cdot\varepsilon^\beta$. No material specific sensitivity threshold can be recognized from this plot. The S parameter begins to increase significantly in the range $0.001\leqslant\varepsilon\leqslant0.01$.

considerations to two annihilation events, namely in deep traps and in the perfect lattice. In this way, the results of the temperature-dependent measurements of the Doppler broadening are presented in terms of the probability $P(T)$ [cf. Eq. (2)] of a positron to annihilate in a deep, vacancylike trap. In this two-state model $P(T)$ is calculated from $S(T)$ by

$$P(T)=\frac{S(T)-S_b(T)}{S_v-S_b(T)}, \qquad (2)$$

where S_v is the value for saturation trapping into deep, vacancylike traps. The temperature-dependent value for annihilation in the perfect lattice $S_b(T)$ was measured on a well-annealed specimen and approximated by a second-order polynomial which provided an appropriate description. This description is sufficient since the concentration of deep traps can be recognized at room temperature by a shift of P. With the known temperature dependence $S_b(T)$ all peculiarities on the temperature dependence of $P(T)$ towards lower temperature can only be caused by dislocations.

S_v is assumed to be independent of temperature and to be about 8% larger than $S_b(T=290$ K$)$. This is in agreement with thermal equilibrium measurements on aluminum and copper which found increases of the S parameter in saturation trapping between 8 and 10 %.[35,36] This rough estimate of S_v is, however, noncritical for our purposes since it mainly affects the absolute value of the $P(T)$ curves and only weakly the features of their temperature dependence. The denominater in Eq. (2) is basically a constant since it varies with temperature just by 5% whereas the variation of the enumerator is much larger. Therefore, the positron-dislocation line binding energy E_b calculated lateron will not be affected by the uncertainty in the estimate of S_v.

A. *In situ* Doppler-broadening measurements during tensile deformation

The *in situ* measurements were performed in the loaded state when straining was interrupted ($\dot{\varepsilon}=0$). The interruption of straining is accompanied with a stress relaxation of a
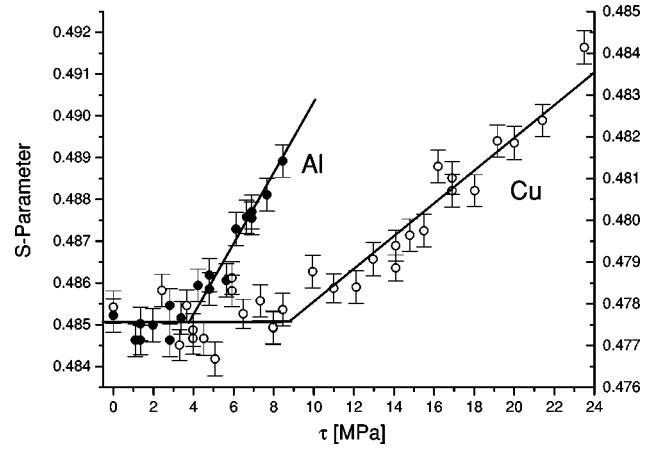

FIG. 3. The S parameter (Ref. 37) presented as a function of the resolved shear stress for copper (Cu-1) and aluminium polycrystals (right and left axis, respectively) demonstrates the existence of a sensitivity threshold of $\tau=(4\pm1)$ MPa in aluminium and $\tau=(9\pm1)$ MPa in copper.

few MPa. The recording of γ-ray spectra was started after the main portion of this stress relaxation was terminated, i.e., some 10 s after straining had been stopped. When tensile straining was continued the amount of relaxed stress was recovered immediately and strain hardening proceeded. At selected stress levels, when intermediate temperature-dependent Doppler-broadening measurements in the cryostat were planed, the mechanical load was completely removed and Doppler measurements were performed at zero stress. For aluminum these tests were performed after stresses of $\tau=1.5$, 3, 5, and 7 MPa had been reached; the corresponding figures for copper are $\tau=4$, 6, 8, 14, and 17 MPa (Fig. 3). A significant difference between the S parameter measured under load and unloaded was not found.

The correlation between the S parameter and the strain ε in Fig. 2 shows no material specific sensitivity threshold. The S parameter starts to increase in the range $0.001\leqslant\varepsilon\leqslant0.01$ in both materials. $S(\varepsilon)$ can be well described by a power law $S(\varepsilon)=S_b+\alpha\cdot\varepsilon^\beta$ with a strongly material-dependent parameter β (see Table I).

Plotting the S parameter versus the resolved shear stress τ in Fig. 3 reveals the existence of different sensitivity thresholds for both materials. Positrons become sensitive to plastic deformation at $\tau=(9\pm1)$ MPa and at $\tau=(4\pm1)$ MPa in copper and aluminum, respectively.

The dependence of S on τ above the threshold can be well described by a linear relation. Above the threshold, the slope $S(\tau)/\tau$ indicates a stronger stress dependence of the S-parameter for aluminum than for copper.

TABLE I. The parameter β in the power law $S(\varepsilon)=S_b$ $+\alpha\cdot\varepsilon^\beta$ depends strongly on the material. The square-root-like behavior of $S(\varepsilon)$ in aluminium is obviously different from the stronger than linear dependence found in copper (specimen Cu-1).

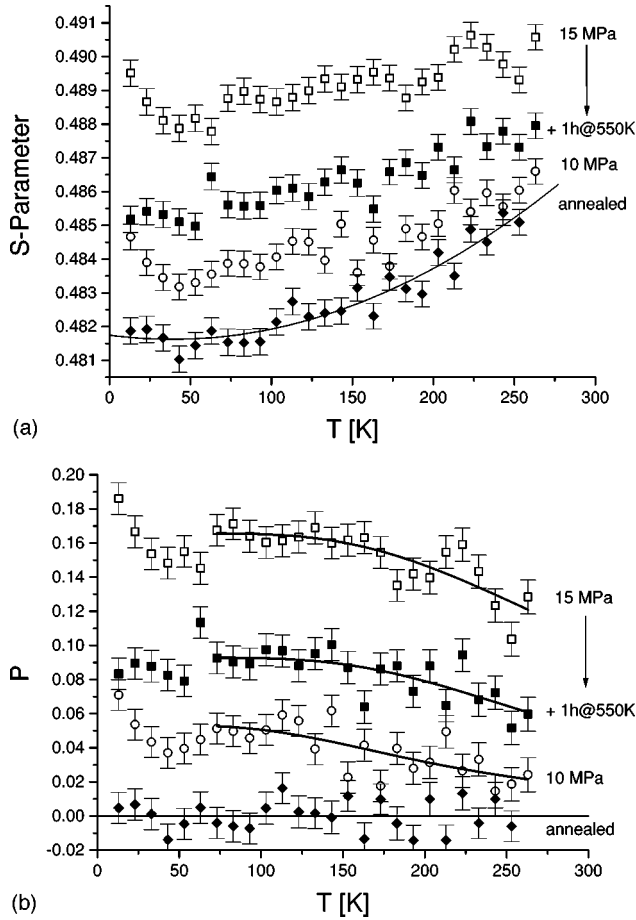| Material	S_b	α	β
Cu	0.4775	1.3 ± 0.8	1.44 ± 0.16
Al	0.4844	0.012 ± 0.002	0.48 ± 0.07

FIG. 4. (a) The temperature dependence of the S parameter is presented for well-annealed copper and after gradual weak tensile deformation up to $\tau = 10$ MPa and up to $\tau = 15$ MPa, the latter followed by an annealing treatment for 1 h at 550 K. Please note that the nondestructive *in situ* technique allows us to take all data from the same specimen (Cu-1). A polynomial was fitted to the well-annealed state which is considered as the behavior of the ideal lattice $S_b(T)$. (b) The corresponding $P(T)$ data are calculated according to Eq. (2); P stands for the probability that positrons annihilate from deep traps. The curves are fits to the trapping model outlined in Sec. IV. The annealing treatment for 1 h at 550 K reduced the magnitude of $P(T)$ achieved after deformation to 15 MPa by approximately 50%.

B. The temperature dependence of positron annihilation in copper after tensile deformation

1. Weak tensile deformation

The probability of positron trapping in deep traps $P(T)$ [Eq. (2)] exhibits a characteristic temperature dependence for deformations in the range from the sensitivity threshold at $\tau = 9$ MPa up to about 20 MPa. This range will be referred to as *weak tensile deformation* of copper (Figs. 4 and 5). The typical temperature dependence consists of an increase of $P(T)$ between 263 and down to about 150 K. Between 150 and 73 K the probability $P(T)$ remains constant before it slightly drops down to about 50 K. Below 50 K another steep increase is found.

Annealing of the specimen deformed to $\tau = 15$ MPa for 1 h at 550 K reduced the magnitude of $P(T)$ by roughly 50% but did not affect the characteristics of the temperature de-
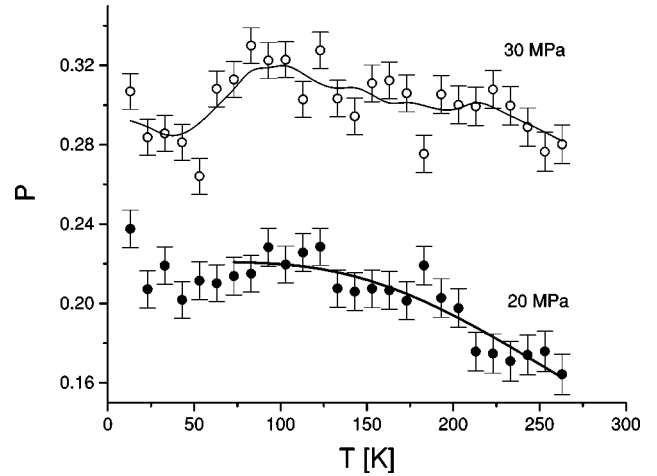

FIG. 5. Comparing the $P(T)$ curves [cf. Eq. (2)] for copper (specimen Cu-1) after tensile deformation to 20 and 30 MPa, one recognizes a change in the characteristics of the temperature dependence. At 30 MPa a significant drop in $P(T)$ below 90 K emerges. The line at $\tau = 20$ MPa presents a fit to the trapping model outlined in Sec. IV. The line drawn for $\tau = 30$ MPa is an eye guide; a fit as for $\tau = 20$ MPa is no longer feasible.

pendence of the curve between 263 and 73 K (Fig. 4). Merely, the very low-temperature increase of $P(T)$ disappeared after annealing.

2. Strong tensile deformation

On copper specimens subjected to strong tensile deformation a distinctly different temperature dependence of the S and P parameters was observed as compared to weak tensile deformation. The transition from weak to strong deformation behavior takes place at $\tau \approx 30$ MPa (Fig. 5). The temperature dependences $S(T)$ and $P(T)$ characteristic for strongly deformed copper exhibit two distinct regions as shown in Fig. 6 for the copper specimen Cu-2 after deformation to $\tau = 53$ MPa. In the first region between 263 and down to 90 K no significant temperature dependence of $P(T)$ can be observed. Below 90 K, however, the second region is characterized by a pronounced decrease of $P(T)$ which continues down to the lowest measured temperature of 13 K.

The effect of annealing on the temperature dependence of $P(T)$ was also studied after strong plastic deformation to $\tau \approx 53$ MPa. The specimen was subjected to a successive series of isochroneous anneals for 1 h starting at 350 K and ending at 650 K with a temperature increment of $\Delta T = 50$ K. From 350 to 550 K $P(T)$ was reduced by 30% without affecting the typical shape of the $P(T)$ curve. After the 600 K anneal a significant reduction of the step height between $P(100$ K$)$ and $P(13$ K$)$ set in which decreased further towards higher annealing temperatures.

In Fig. 6 a feature becomes apparent after the 650 K anneal. The increase in $P(T)$, previously noticed after weak deformation, between 263 and 150 K reappeared.

C. The temperature dependence of positron annihilation in aluminum after tensile deformation

The temperature dependence of $P(T)$ observed in the aluminum specimen after tensile deformation up to a resolved
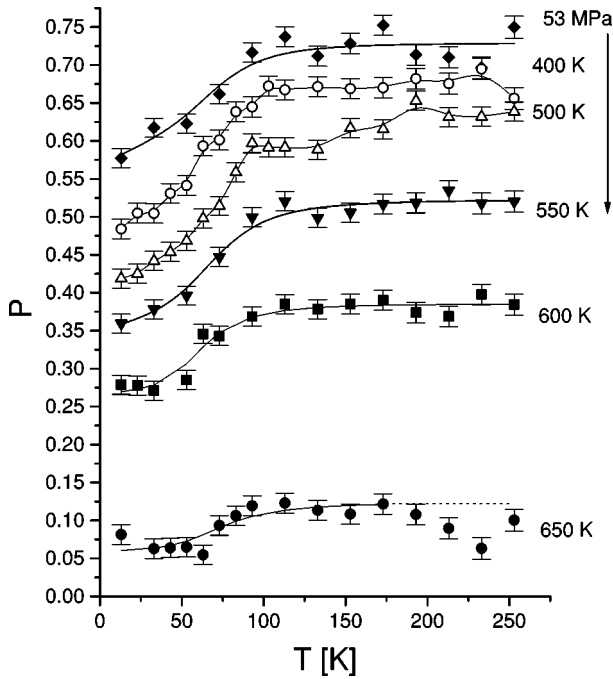
FIG. 6. The characteristic temperature dependence of the $P(T)$ curve of strongly deformed copper (specimen Cu-2; $\tau=53$ MPa) resists isochronous annealing treatments for 1 h up to temperatures of 550 K. The curves are only shifted to lower $P(T)$ values. After 600 K annealing the low-temperature drop of $P(T)$ begins to flatten. After 1 h at 650 K the increase in $P(T)$, noted before in the weakly deformed specimen between 263 and 150 K, appears again. The lines drawn for 400 and 500 K are eye guides, whereas all other curves are fits to the model outlined in Sec. IV.

shear stress of $\tau=4.8$ MPa and $\tau=6.9$ MPa is depicted in Fig. 7. The $P(T)$ curve is basically flat over the temperature range from 263 down to 70 K. Below 70 K it increases sharply. With progressing deformation this increase at low temperatures becomes more accentuated. In contrast to copper, no differences between weakly and strongly deformed conditions can be recognized at least in the deformation range covered by our experiments.

IV. TRAPPING MODEL

The quantitative analysis of the data presented above requires the assumption of a specific model. We employed a three-state trapping model (Fig. 8) which is frequently used to describe the observed temperature dependence.[18,24,29,38]

The model is based on the following assumptions: The possible three states of the positron are the perfect lattice, the dislocation line, and a vacancylike defect. In agreement with nearly all reliable experimental results (e.g., Refs. 19,24) and theoretical considerations[20,29,39] the dislocation line is considered as a shallow trap with a binding energy of E_b $\lesssim 0.1$ eV. The bulk and true dislocation lines, i.e., without jogs or associated vacancies, have very similar annihilation characteristics which cannot reliably be separated. The peculiar role dislocation lines play for positron trapping is that they may act as precursor states for transitions into deeper traps such as jogs or vacancies in their close vicinity. The transition rate η into these deep *associated* defects and that of direct trapping in vacancies η' are independent of
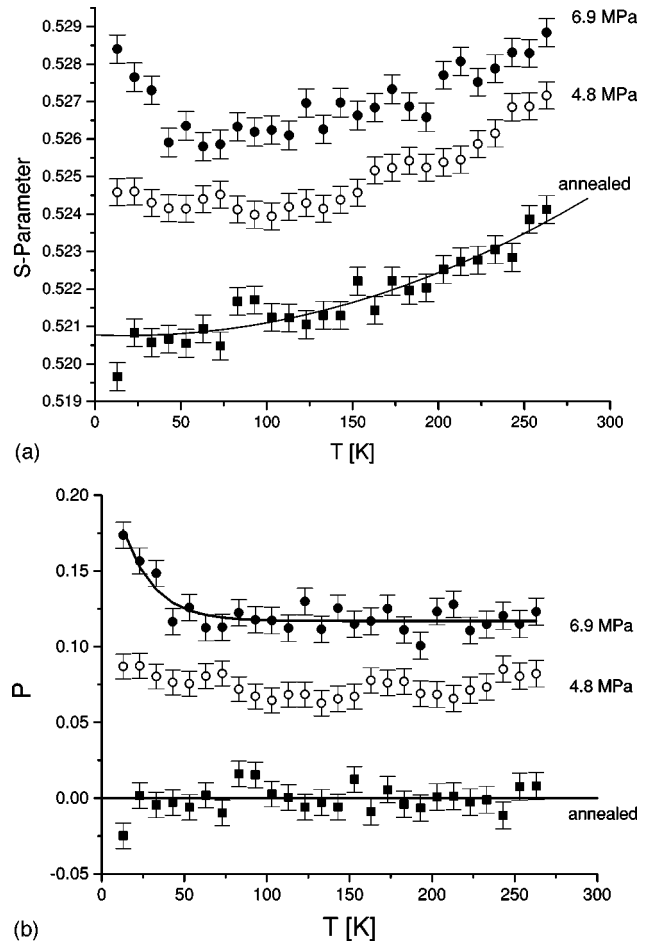

FIG. 7. (a) The temperature dependence of the S parameter in the aluminium specimen in the well-annealed state and after successive gradual tensile deformation up to 4.8 and 6.9 MPa. After deformation to 6.9 MPa a marked increase of S is observable below $T=70$ K. The annealed state is approximated by a second-order polynomial which reflects the temperature dependence in the bulk, $S_b(T)$. (b) The corresponding $P(T)$ curves are calculated according to Eq. (2). The solid curves result from a fit to the trapping model outlined in Sec. IV.
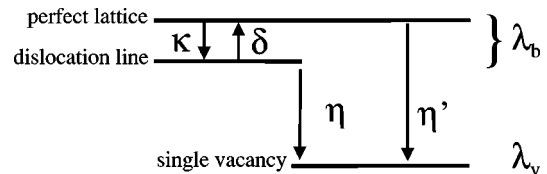
FIG. 8. The trapping model applied to interpret the above results assumes three states, namely, the perfect lattice, the dislocation line and a vacancylike defect. The transition rates from the perfect lattice to the dislocation line and directly to the vacancylike defect are denoted by κ and η', respectively. Positrons may escape from the dislocation line into the lattice with the detrapping rate δ or undergo a transition into a deeper, vacancylike trap with the rate η. Since the electronic environment of a positron in the perfect lattice and along a dislocation line is very similar, approximately the same decay rate λ_b can be assumed. The annihilation rate in a vacancy is denoted λ_v.

TABLE II. Fitting parameter for aluminium and weakly deformed copper (specimen Cu-1). The dislocation density has been calculated according to Eq. (10). Parameters marked with * were equated to zero, assuming temperature-independent trapping (ν_0) or negligible direct trapping into lattice vacancies (η').

Specimen	ν_1 (10^{-4} m^2/s)	ν_0 (10^{-4} m^2/s)	ϱ (10^{12}/m^2)	E_b (meV)	η (1/ps)	η' (1/ns)
Al 6.9 MPa	1.3 ± 0.03	1.7 ± 0.5	6	36 ± 0.3		* 0
Cu 10 MPa	1.2 ± 0.2	* 0	4.3	26 ± 30	0.9 ± 2	* 0
Cu 15 MPa	1.5 ± 0.05	* 0	12	47 ± 28	1.2 ± 2	* 0
+ 1h@550 K	0.8 ± 0.4	* 0	12	50 ± 40	0.4 ± 0.9	* 0
Cu 20 MPa	1.3 ± 0.03	* 0	20	39 ± 16	1.6 ± 1.6	* 0

temperature.[40,41] For equal concentrations of defects, the transition rate η into dislocation associated defects is, however, higher than the rate η' for direct vacancy trapping.[29]

Since the annihilation behavior of positrons in the perfect lattice and along ideal dislocation lines on one side, and in vacancies or in jogs on the other side is so similar, the experiment can only distinguish between positrons annihilating from deep traps and others. Only two decay rates λ_b and λ_v are accessible. The model can therefore be simplified to a two-state model in which the probability $P(T)$ for positrons to annihilate in a deep trap can be expressed in terms of the transition rates as[29]

$$P(T) = \frac{\eta'[\lambda_b + \delta(T) + \eta] + \eta\kappa(T)}{[\lambda_b + \kappa(T) + \eta'](\lambda_b + \eta) + \delta(T)(\lambda_b + \eta')}. \quad (3)$$

The temperature dependence of $P(T)$ is caused by the trapping rate in the dislocation-line state $\kappa(T)$, and the detrapping rate out of this state $\delta(T)$. Numerical expressions were given for both $\kappa(T)$ and $\delta(T)$ in Refs. 19,29. There, the trapping rate at dislocation lines

$$\kappa(T) = \varrho\,\nu(T) \quad (4)$$

depends on the dislocation density ϱ and the specific trapping rate at dislocations which can be approximated by

$$\nu(T) = \nu_1 + \nu_0 \exp\left\{-\frac{k_B T}{2(k_B\Theta_D - E_b)}\right\}, \quad (5)$$

where k_B denotes the Boltzmann constant, Θ_D the Debye temperature and E_b the binding energy of the positron to the dislocation line, i.e., the depth of the shallow trap. The strong temperature dependence of the detrapping rate is given by

$$\delta(T) = C_0\nu(T)k_B T\sqrt{\frac{k_B T}{E_b}}\exp\left\{-\frac{E_b}{k_B T}\right\} \quad (6)$$

and the constant

$$C_0 = \frac{m_e^*}{4\sqrt{\pi}\hbar^2}. \quad (7)$$

C_0 depends on the effective positron mass m_e^* which is equated with the rest mass of the electron.

The transition of the positron into shallow traps such as dislocation lines is realized by the emission of acoustic phonons if the energy of the positron E_p fulfills the condition $E_p < k_B\Theta_D - E_b$. For deeper traps electron hole pair excita-

tions become important for the positron trapping process. It has to be stressed that the expression for $\nu(T)$ proposed by Petersen et al.[19] is an approximation which assumes $E_b < k_B\Theta_D$. Smedskajaer et al.[29] used a similar exponential term for $\nu(T)$ but without specifying the relation between the exponent and E_b.

Before fitting the above model to the $P(T)$ data, we made some reasonable assumptions which allowed us to simplify Eq. (3). For aluminum and weakly deformed copper it is justified to assume $\eta \gg \lambda_b$ and $\eta' = 0$. This means that positrons which are already localized along dislocation lines will be trapped in associated defects and that direct trapping in single vacancies in the perfect lattice is negligible. These assumptions seem reasonable since vacancies in aluminum are mobile at room temperature[42] and their concentration is still low in weakly deformed copper. Eq. (3) can then be transformed into

$$P(T) \approx \frac{\kappa(T)\eta/[\eta + \delta(T)]}{\lambda_b + \kappa(T)\eta/[\eta + \delta(T)]}. \quad (8)$$

The direct trapping in vacancies cannot be neglected in strongly deformed copper where $\eta' \geq \lambda_b$. Hence we employed Eq. (3) for the numerical analysis.

In deformed aluminum no effect of $\delta(T)$ was noticed which let us conclude that $\eta \gg \delta(T)$ [cf. Eq. (8)]. For the purpose of fitting the aluminum data we therefore simplified Eq. (8) to

$$P(T) \approx \frac{\kappa(T)}{\lambda_b + \kappa(T)}. \quad (9)$$

Tables II and III compile the values which can be attributed to the parameters of the trapping model after a numerical analysis of the $P(T)$ curves. The dislocation density ϱ was estimated from the resolved shear stresses τ via the relation

$$\varrho = \left(\frac{\tau}{\alpha b G}\right)^2, \quad (10)$$

where $0.3 \leq \alpha \leq 0.5$ is a numerical factor and b and G denote the modulus of the Burgers vector and the shear modulus, respectively.[43] The numerical constants used for the calculations are listed in Table IV.[46–48]

For the $P(T)$ curves obtained on strongly deformed copper η had to be set to 0 to achieve satisfactory fits. This means that the transition from dislocation lines into associ-

TABLE III. Fitting parameter for strongly deformed copper (specimen Cu-2) with subsequent annealing treatments. Best fits were obtained when η was equated to zero, i.e, suppressing a transition of positrons trapped along dislocation lines into deeper traps. This transition is effectively suppressed for $\eta < 1$/ns.

Specimen	ν_0 $(10^{-4}$ m^2/s)	ϱ $(10^{13}$/m$^2)$	E_b (meV)	η (1/ps)	η' (1/ns)
Cu 53 MPa	1.3 ± 1.1	8 ± 6	25 ± 4	0	25 ± 1
+ 1 h @ 350–550 K	1.0 ± 0.9	10 ± 8	23 ± 6	0	10.0 ± 0.2
+ 1 h @ 600 K	1 ± 3	7 ± 18	21 ± 15	0	5.7 ± 0.2

ated deeper defects has to be suppressed to account for the decline in $P(T)$ towards lower temperature.

V. DISCUSSION

A. Sensitivity threshold of positron annihilation to dislocations

Figure 9 shows the resolved shear stress versus strain for well-annealed aluminum and copper. The sensitivity threshold of positron annihilation to the defects created by plastic deformation is indicated for both materials. These threshold values were obtained from an interpolation of the S parameter data in Fig. 3 under the assumption of constant S parameter at low resolved shear stresses and a linear increase at higher values.

In order to determine the sensitivity limit of positrons to plastic deformation it proved necessary to carefully perform tensile tests and to interupt them after small strain increments in order to perform positron-annihilation measurements. The applied *in situ* technique is nondestructive and allowed to follow the development of the Doppler broadening in a single specimen during the microstructure evolution which manifests itself in the tensile hardening curve τ versus ε. Moreover, the specimen had not to be dismounted from the test machine apart from the temperature-dependent measurements in the cryostat. Thus, the risk of unintentional and uncontrolled deformation of the specimen during dismounting and remounting could be reduced to a minimum. Soft, weakly deformed specimens are especially susceptible to such deformation during handling. The problem is less pronounced in specimens which have already undergone some tensile hardening. The existence of a sensitivity threshold was found earlier in copper single crystals.[16] The relation between the mean diffusion length $L_+ = \sqrt{6D_+ / \lambda_b}$ and the mean dislocation spacing d at the threshold

$$d = 2 \cdot L_+ \qquad (11)$$

was found to be valid for polycrystalline copper as well as for polycrystalline aluminum. Table V shows the values obtained by Eqs. (10) and (11). The material constants used are compiled in Table IV. Equation (11) relates the positron diffusion length in the perfect material to the dislocation density at the sensitivity threshold. If this relation can be verified for further materials it may serve as a rule of thumb to estimate the degree of deformation at which positrons become sensitive to dislocations. The sensitivity threshold of (9 ± 1) MPa for polycrystalline copper agrees well with the (10 ± 1) MPa obtained in an earlier investigation by *in situ* positron-lifetime measurements on copper single crystals.[16]

The observation that this relation holds in aluminum and in copper is a strong indication that dislocations indeed play an important role as trapping sites for positrons even at room temperature. It has been estimated that the concentration of vacancies present in specimens up to the sensitivity threshold is still below their detection limit.[16] Copper deformed close to the sensitivity threshold (e.g., $\tau = 10$ MPa, Fig. 4) cannot be differentiated from the well-annealed state at room temperature, whereas at 13 K the S parameter for the deformed state is significantly higher. This can be explained by the reduction of the ratio $\delta(T)/\kappa(T)$ between detrapping from dislocation lines and trapping along them. Thus, the detection limit has to be regarded as a function of temperature. If the detrapping rate of positrons from dislocation lines is smaller at lower temperatures even smaller dislocation densities become detectable.

The fact that we did not find a significant difference between the Doppler-broadening measurements in the loaded and the unloaded state of the aluminum and the copper specimen caused us to have a closer lock on the work of Iwami *et al.*[44] These authors found a small but measurable effect in 8% cold-rolled aluminum polycrystals after annealing at 108 °C for 1 h. For example, an increase of the positron lifetime in the trapped state of 7 ps was reported after applying a stress of only 0.26 MPa. In our case, such an effect would correspond to the extent of the uncertainty of our S-parameter measurements and would hence be difficult to observe. On the other hand, our aluminum polycrystal was unloaded from resolved shear stresses up to 7 MPa. The microstructure obtained by tensile deformation is, however, different from the conditions established by Iwami *et al.*[44] after cold rolling to a thickness reduction of 8% and subsequent annealing. The dislocation density in our specimens is presumably much lower and the remaining internal stresses after unloading are much higher than in the samples of Iwami *et al.*[44] Moreover, in our *in situ* positron-annihilation measurements the annealing of atomic defects is still incomplete.

TABLE IV. Material constants of copper and aluminium used for the calculations.

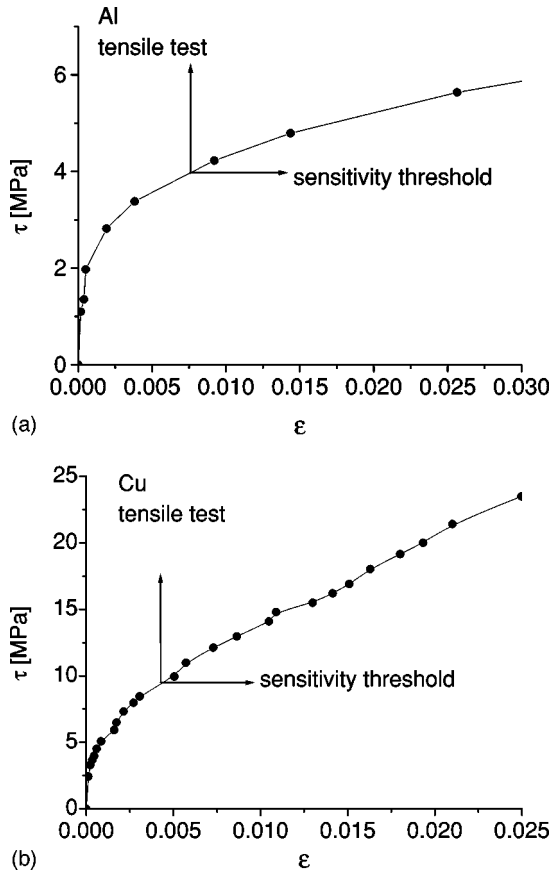| Material	λ_b (1/ns)	α	b $(10^{-10}$ m$)$	G (MPa)	D_+ (cm^2/s)	Θ_D (K)
Cu	9.1	0.4	2.56	46000	1.0	343
Al	6	0.4	2.86	25000	1.0	428
Reference	10	43	46	46	9,47	48

FIG. 9. Resolved shear stress τ versus strain ε curves for (a) aluminium and (b) copper (specimen Cu-1) with the sensitivity limits marked.

Another noteworthy feature found in the *in situ* experiments is the material specific parameter β obtained by fitting a power law to the $S(\varepsilon)$ curves (Fig. 2). Aluminum and copper mainly differ in the mobility of vacancies at room temperature and the core structure of dislocations. Both determine the kind of defects which will remain after plastic deformation. Vacancies created by dragging of jogs on moving screw dislocations anneal out at room temperature much faster in aluminum than in copper. The power β, which describes the increase of the S parameter with proceeding deformation, is expected to be sensitive to the efficiency of the production of positron traps. Or in other words, β is a measure for the accumulation rate of vancancylike defects in the material.

B. Temperature dependence of $P(T)$

1. Trapping in vacancies

In copper the temperature range 350–550 K corresponds to the annealing stages III and IV where vacancies are mobile and vanish in sinks such as dislocation lines or grain boundaries.[42] In the case of high vacancy concentrations also stable clusters or dislocation loops can be formed. The annealing study of the strongly deformed copper specimen showed clearly that the trapping rate in vacancies η' does not depend on temperature. The reduction of the vacancy concentration by annealing shifts the $P(T)$ curve in Fig. 6 to lower values without changing its typical features. Even the

TABLE V. The experimentally determined sensitivity thresholds $\tau_{\text{threshold}}$ and their corresponding dislocation densities ϱ, calculated according to Eq. (10), are related to the mean dislocation spacing d via $d = 1/\varrho^{0.5}$. Twice the mean diffusion length L_+ equals approximately d.

Material	$\tau_{\text{threshold}}$ (MPa)	ϱ (m^{-2})	d (nm)	$2 \times L_+$ (nm)
Cu	9 ± 1	3×10^{12}	500 ± 60	510
Al	4 ± 1	2×10^{12}	710 ± 180	630

absolute difference $P(250\ \text{K}) - P(13\ \text{K}) \approx 0.15$ is preserved up to the isochronal annealing step at 550 K. The decrease of this value during the subsequent annealing steps at higher temperatures can be explained by the recovery of dislocations. Therefore, we conclude that the contribution of vacancies to $P(T)$ is a constant and, consequently, η' is also constant with respect to temperature.

2. Trapping at dislocations after weak deformation

The temperature dependence observed in weakly deformed copper can be ascribed to the reduction of the detrapping $\delta(T)$ from dislocation lines with decreasing temperature. This leads to the increased probability that positrons are localized along dislocation lines and that they will undergo a subsequent transition into associated deeper traps. The fitting range was restricted to temperatures $T \geq 70$ K where $\kappa(T)$ can be regarded as weakly temperature dependent.[29] Consequently, $\nu(T) = \nu_1$ was assumed to be constant. The resulting binding energies E_b decrease with proceeding deformation from (50 ± 40) meV to (39 ± 16) meV determined at $\tau = 15$ MPa (and 1 h of annealing at 550 K) and at $\tau = 20$ MPa, respectively. The ideally well-separated dislocation lines, which are present in well-annealed specimens, multiply and tend to cluster during deformation due to their long-range elastic interaction. A large variety of different positron binding energies to dislocations may arise from those interactions. The effective potential seen by positrons may decrease because they do not necessarily have to escape into the ideal, undistorted lattice. They may also escape into an environment with somehow modified atom positions due to the elastic stress and strain fields around other nearby dislocations. Therefore, we may consider the positron-dislocation binding energy obtained for the lowest observable dislocation density as the most reliable one. Consequently, positrons have a binding energy of $E_b \geq 50$ meV to dislocations in copper. This value agrees well with the results of molecular-dynamics simulations of a single dissociated edge dislocation in copper which yielded a binding energy of $E_b = 60$ meV.[20]

The energy transfer necessary for a transition from the perfect lattice to a dislocation line cannot be achieved solely by the emission of a single phonon since the binding energies E_b are larger than $k_B \Theta_D(Cu) \approx 30$ meV. According to Smedskjaer *et al.*[29] this yields a weaker temperature dependence of the specific trapping rate $\nu(T)$ and supports our previous approximation that $\nu(T)$ is constant in the considered temperature range.

The dislocation density in the specimen Cu-1 deformed to 15 MPa was only slightly affected by the annealing treatment

for 1 h at 550 K (Fig. 4). However, the whole $P(T)$ curve is shifted to lower values. This shows that thermally activated, mobile defects contribute significantly to positron trapping in this stage of deformation. These defects can be single vacancies as well as jogs which may vanish both at elevated temperatures due to the annihilation of vacancies along dislocation lines. The reduced jog concentration may also be responsible for the flattening of the $P(T)$ curve at temperatures $T<50$ K. The increasing ratio of $\kappa(T)/\delta(T)$ towards lower temperatures may become visible only if the dislocations can guide the positrons trapped along them to deeper traps associated with them. Otherwise positrons annihilate directly from dislocation lines which also contributes to the decrease of $P(T)$.

The temperature dependence found in aluminum is much simpler. The only feature the $P(T)$ curve exhibits is an increase below 70 K which becomes more pronounced with proceeding deformation. This effect is attributed to enhanced trapping $\kappa(T)$ to dislocation lines at low temperatures. The obtained positron-dislocation binding energy of about (36 ± 1) meV is in reasonable agreement with 31 meV of Petersen et al.[19]. E_b is smaller than in weakly deformed copper but no effect of an increased detrapping δ could be recognized which would be visible in Fig. 7 by a reduction of $P(T)$ towards higher temperature. This may tentatively be explained by a much faster transition η of the positrons from dislocation lines into associated deeper traps. If η is large in comparison to the detrapping δ, the reduction of δ towards lower temperature may not be relevant [cf. Eqs. (8) and (9)]. A cogitable explanation is the different dislocation structure owing to different stacking fault energies. The widely dissociated dislocations in copper seem to hinder the transition into jogs or other associated deep traps, whereas the narrow dislocations in aluminum facilitate it, provided that the jog density is approximately the same.

The absence of detrapping in aluminum might also be explained by assuming an appreciably higher jog density in aluminum than in copper. For the narrow dislocations in aluminum, cross slip is a much easier process than for dissociated dislocations in copper. In this way screw dislocations in aluminum might contain a higher density of jogs as cross slip traces than screw dislocations in copper. Moreover, the dragging of jogs by moving screw dislocations yields the production of vacancies and interstitials which can easily migrate to sinks like grain boundaries and dislocations. The absorption of atomic defects along dislocation lines may lead to the formation of additional monoatomic jogs but may also annihilate existing ones. Due to this complex behavior it is not clear whether the jog density along dislocations in aluminum may indeed be larger than in copper. Unfortunately, there are no convincing experimental methods which are selectively sensitive to monoatomic jogs.

However, the obtained $E_b=(36\pm1)$ meV is very close to $k_B\Theta_D(\text{Al})\approx37$ meV and we feel prompted to be very careful with an interpretation of this value. Equation (5) for $\nu(T)$ is an approximation which surely becomes critical close to the Debye cutoff at $E_b=k_B\Theta_D$. It is interesting to note that ν_1 and ν_0 are approximately equal. From this feature one can recognize that in aluminum a temperature-independent process cannot be neglected for the transition of positrons to dislocation lines. As known from theoretical considerations, electron hole pair excitations do not depend on temperature.[28] They become increasingly important for the transition process if the binding energy to the dislocation line is larger than $k_B\Theta_D$. The binding energy is likely to be underestimated by using expression (5) due to the restriction that $E_b<k_B\Theta_D$. One might argue that the real positron dislocation binding energy in aluminum should be at least of the order of that in copper, considering the less dissociated dislocations with a more localized dilatational zone.

The specific trapping rates ν_0 and ν_1 are similar in copper and in aluminum and lie between 0.8 and 1.7 cm^2/s. They are larger than the theoretical estimates[29] but agree well with experimental values of Iwami et al.[44] which range from 1 to 4.5 cm^2/s. These authors obtained the highest value for their lowest observed dislocation density at about 10^{12} m^{-2}. The specific trapping rate was found to decrease with increasing dislocation density.

In spite of the uncertainties for the transition rate η from shallow dislocation lines into deeper traps, the values obtained from our curves in the case of weakly deformed copper are very large. They are of the order of $\eta\approx10^{12}$s^{-1} which is, as assumed before, much higher than the annihilation rate λ_b.

3. Trapping at dislocations after strong deformation

The striking difference exhibited by $P(T)$ for weakly and strongly deformed copper raises the question whether these two effects can be related to positron trapping at dislocations alone or if a new type of trap has to be introduced to account for the observations.

A slight drop in the $P(T)$ curves below 75 K can already be observed in copper after deformation up to 10 MPa and to 15 MPa. This means that the responsible defects are already created by weak deformation, but they do not significantly dominate the temperature dependence until deformation is continued up to 30 MPa. The annealing experiment (Fig. 6) performed after tensile deformation up to 53 MPa demonstrated that this type of trap is stable at high temperatures and showed that its Doppler-broadening signature is very similar to that of the perfect lattice. This can be concluded from the fact that after the 650 K anneal the decrease in $P(T)$ at low temperatures is still present, although the whole curve is close to the one in the annealed state with $P(T)=0$. Hence, it seems reasonable to identify this trap with a dislocation line or dislocation arrangement. The binding energy of $E_b=(25\pm4)$ meV is sufficient to trap positrons at low temperatures in a way that the transition to deeper traps is hindered ($\eta<1/\text{ns}$). The mobility $M_D^{e^+}$ of the positrons along a dislocation line may be the clue to an explanation in this case. $M_D^{e^+}$ is surely determined by the atomic configuration of the dislocation core. Along perfect nondissociated (''textbook'') dislocations the movement of positrons may easily be possible, whereas along dissociated dislocations it might be controled and hindered by the more complicated arrangement of the atoms in the stacking fault ribbons and in the cores of the remaining partial dislocations. Intuitively, it appears to be clear that the picture of a channel for roughly unhindered migration becomes invalid if the dislocation dissociates into partials.

Thus, positrons may get trapped along dissociated dislocation lines at low temperatures which prevent them from reaching deeper traps. This effect is clearly visible only after high degrees of deformation when the concentration of vacancylike defects, such as jogs, is so high that direct trapping η' in these defects dominates. Figure 6 shows that $P(T)$ of copper after deformation to 53 MPa is constant down to 100 K before the fraction of positrons annihilating in deep traps diminishes. The increasing portion of positrons which gets trapped at dissociated dislocations and annihilates before a transition into deeper defects takes place leads to a noticeable reduction of the Doppler broadening. However, it is not clear whether the observed temperature dependence of $P(T)$ is indeed caused by the trapping $\nu(T)$ and detrapping $\delta(T)$ at dislocation lines or if the mobility of positrons along dislocation lines $M_D^{e^+}$ has also to be considered.

This problem might be tackled by slow positron-beam experiments. If positrons do diffuse along dislocation lines more easily than through the perfect lattice then the backdiffusion probability to the surface of a material should be increased if dislocations reach the surface. One should compare the back diffusion of positrons implanted to the same depth in weakly tensile deformed copper and aluminum single crystals with an identical dislocation density and identical orientation of the crystal lattice towards the incident positron beam. Such experiments may elucidate the role of dislocations as diffusion paths for positrons. The considerations above would be supported if the efficiency of back diffusion is higher in aluminum than in copper.

Further insight on the positron dislocation interaction can be gained by high-resolution positron microscopy.[45] Positron beam diameters of 1 μm offer the possibility of studying directly dislocation bundles or even single dislocation lines. This may help clarify the influence of the dislocation distribution on the trapping of positrons.

VI. SUMMARY AND CONCLUSIONS

In order to unravel the interaction between positrons and the complicated defect spectra produced by plastic deformation, temperature-dependent positron-annihilation measurements have been performed after selected degrees of plastic deformation. Recently developed techniques of *in situ* positron-annihilation measurements during tensile deformation are helpful to chose the appropriate degrees of deformation.[16] Moreover, a complete series of measurements can be performed on a single specimen to observe the effects of the microstructure evolution during tensile tests. This reduces the scatter which may appear if different stages of microstructure evolution have to be investigated in different specimens.

The *in situ* techniques revealed a sensitivity threshold of positron annihilation to plastic deformation damage which can be correlated with a critical dislocation density. As a rule of thumb, the threshold is given by Eq. (11). This relation was found on polycrystalline aluminum and copper and earlier by *in situ* positron-lifetime measurements on copper single crystals.[16] The *in situ* techniques also enable a comparison of the efficiency of positron-trap formation during plastic deformation of different materials.

In combination with isochronal annealing experiments further information on the defect-type acting as positron trap can be obtained. However, the effect of annealing of vacancies on the trapping behavior of dislocations is extremely difficult to control. Thus, experiments close to the sensitivity threshold where the content of vacancies is still low[16] were performed.

The present experiments revealed a different behavior of copper after weak and strong plastic deformation. After strong plastic deformation, i.e., to a resolved shear stress above 30 MPa, the transition of positrons trapped along dislocation lines into deeper traps appears to be suppressed. A similar behavior has not been observed in aluminum.

A quantitative evaluation of the data by help of a three-state trapping model yielded binding energies of positrons to dislocation lines in copper and aluminum of $E_b \geqslant 50$ meV and $E_b = 36$ meV, respectively. The derived specific trapping rates into dislocations are 0.8 cm^2/s and 1.7 cm^2/s, respectively. The transition rates from dislocation lines into deeper traps associated with the dislocations are of the order 10^{12} s^{-1}. In strongly deformed copper, however, this rate had to be equated with zero in order to obtain reasonable fits of the model to the data.

Future experiments will be performed on other face-centered-cubic materials like nickel and especially on body-centered-cubic materials like α-iron and molybdenum. The latter allow one to distinguish between screw and edge dislocation by the proper choice of the deformation mode and temperature and to study effects of the spatial dislocation distribution and the dislocation type on the sensitivity threshold.

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