Temperature dependence of the rate of positron trapping by vacancies in gold*

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Positron lifetimes were measured in high-purity gold in order to study the temperature dependence of the monovacancy-positron specific trapping rate, defined as the positron trapping rate per unit monovacancy concentration. In all cases a positive temperature dependence was found. Equilibrium experiments alone yielded a temperature dependence of $T^{0.5\pm0.2}$ over the temperature range 673–1033 K. Experiments which combined quenching and equilibrium measurements indicated a temperature dependence of $T^{0.9\pm0.1}$ between 243 and 1033 K. Additional measurements at lower temperatures indicated that a positive temperature dependence continues down to 115 K.

I. INTRODUCTION

Positron annihilation holds much promise as a technique for measuring vacancy concentrations in metals. According to the trapping model,[1] which describes the behavior of positrons in metals containing vacancies, the monovacancy-positron trapping rate $\kappa_{1V}(T)$ is given by a constant times the monovacancy concentration, i.e.,

$$\kappa_{1V}(T) = \mu_{1V}(T)\, C_{1V}(T). \tag{1}$$

Recently, there has been a growing interest in the temperature dependence of the parameter μ_{1V}, which we call the monovacancy-positron specific trapping rate. The temperature dependence of μ_{1V} must be known if the monovacancy-formation energy E^F_{1V} is to be deduced from measured values of the positron trapping rate, using the relation

$$\kappa_{1V}(T) = \mu_{1V}(T)\, e^{S^F_{1V}/k}\, e^{-E^F_{1V}/kT},$$

where S^F_{1V} is the monovacancy entropy of formation.

At least three possible temperature dependences for the specific trapping rate have been proposed on theoretical grounds. Several groups[2] have suggested that the specific trapping rate is proportional to the thermal velocity of the positron, which leads to the prediction that μ_{1V} increases as $T^{1/2}$. A second argument, based on the assumption that the positron behaves as an extended wave packet, predicts that μ_{1V} is temperature independent.[3] A third proposal, based on positron-phonon scattering arguments, predicts a $T^{-1/2}$ dependence.[4]

Most often, for simplicity, experimental results have been analyzed assuming that the specific trapping rate is temperature independent.[5] Previously reported experimental evidence concerning the temperature dependence of μ_{1V} has been inconclusive. Connors, Crisp, and West[6] have reported that experiments on cadmium support a $T^{-1/2}$ dependence but then have suggested that this may have been an artifact of their experimental procedure. McKee, Stewart, and Jamieson[7] have performed experiments on quenched gold which indicated that there is a negligible temperature dependence in the specific trapping rate between liquid-nitrogen temperature and ice temperature. Recent experiments by Petersen, Thrane, and Trumpy[8] on voids in molybdenum and aluminum indicate a positive temperature dependence in the void-positron specific trapping rate.

In this article, we report the results of two types of experiments on gold, which were performed to determine the temperature dependence of the specific trapping rate.[9] In the first type of experiment, positron lifetimes were measured in gold specimens which were heated to temperatures in the range 673–1033 K, in order to induce a series of thermal-equilibrium vacancy concentrations. From these measurements, we deduced the temperature dependence in the specific trapping rate over the temperature range 673–1033 K, as well as the value of the specific trapping rate at 1033 K. In the second type of experiment, the specific trapping rate was determined at low temperatures in gold specimens which had been quenched from relatively high temperatures in order to freeze in a supersaturation of primarily monovacancies and divacancies. The absolute vacancy concentrations quenched in were determined from quantitative electron-microscopy studies. We will first discuss the equilibrium experiments and then the

quenching experiments.

II. EXPERIMENTS AND RESULTS

The experimental methods and the least-squares analysis of the data for the equilibrium measurements are described in detail in Refs. 1, 10, and 11. Positron-lifetime measurements were made on specimens of 99.9999% gold which was heated to temperatures between 400 and 760 °C. The data were least-squares analyzed using a relatively sophisticated fitting program which had the following features: (a) Data from all temperatures were analyzed simultaneously to eliminate incorrect assumptions concerning the positron lifetime in the lattice and in vacancy traps and to allow for the possibility of temperature independent traps; (b) an exponential-sided resolution function, which closely matches the experimentally observed resolution function, was used throughout the data analysis; (c) the source component was adjusted during the least-squares analysis; and (d) temperature dependence in the specific trapping rate of the form T^x could be allowed for. Reference 1 contains a more detailed discussion of the data-analysis program and of the results of the experiments on gold in thermal equilibrium. Those results which are relevant to the present discussion may be summarized as follows: When the specific trapping rate was assumed to be temperature independent, the trapping rate at 1033 K was found to be $\kappa_{1V} = (24 \pm 2) \times 10^9$ sec^{-1}. When temperature dependence in the specific trapping rate of the form T^x was allowed, an improved fit to the data from measurements between 673 and 1033 K was found for a temperature dependence of $T^{0.5 \pm 0.2}$. In this case, the trapping rate of 1033 K was $\kappa_{1V} = (26 \pm 2) \times 10^9$ sec^{-1}.

The specific trapping rate, μ_{1V}, at 1033 K can be deduced from these results using Eq. (1). The value of the monovacancy concentration at 1033 K is taken to be $C_{1V} = (6.2 \pm 0.2) \times 10^{-5}$ which, within its uncertainties, is consistent with variety of quenching and simultaneous length change and lattice-parameter data.[12] Using $\kappa_{1V} = 26 \times 10^9$ sec^{-1}, we obtain for the specific trapping rate at 1033 K $\mu_{1V} = (42 \pm 5) \times 10^{13}$ sec^{-1}. Here the variation of κ_{1V} with the temperature-dependence parameter x has been absorbed in the uncertainty in μ_{1V}. Thus from the equilibrium experiments, we have obtained the least-squares estimate of the temperature-dependence parameter of the specific trapping rate μ_{1V} at 1033 K.

Several experiments were performed on quenched gold. The specimens for these experiments were 0.15-mm-thick by 5-mm-wide strips of 99.999% gold obtained from Cominco American, Inc. In the first of the experiments, as shown in Fig. 1, the specimen strip was annealed in air at 1203 K for approximately 1 h to remove impurities, equilibrated at ~1133 K, and then quenched edgewise into water at approximately 280 K.

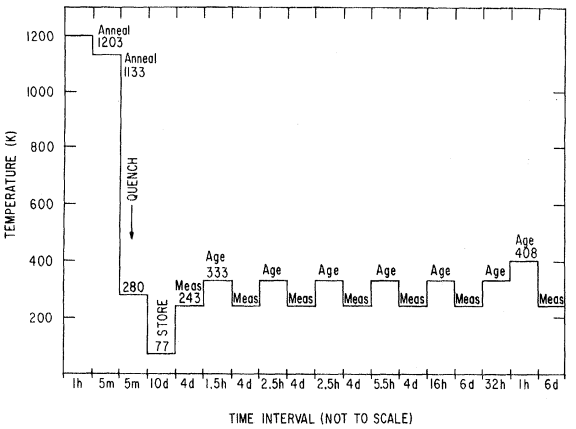

FIG. 1. Heat-treatment sequence for the positron-annihilation sample Au-1. Positron-lifetime measurements were made at 243 K as shown. Electron-microscopy measurements were made on samples which were aged in the same sequence omitting the intervals at 243 K.

After the quench, the specimen strip was cut into 5-mm squares for the positron experiments and into smaller pieces for the electron-microscopy measurements. The electron-microscopy samples were aged at a temperature of 333 K for 1.5, 4.0, 6.5, 12.0, and 28.0 h, respectively; an additional sample was aged for 60 h, with a final aging at 408 K for 1 h. The positron-annihilation sample was aged successively for these times. Positron-lifetime measurements and electron-microscopy measurements were performed on the respective samples between the steps in the aging as shown in Fig. 1. In the course of this aging, the vacancies quenched into the specimen gradually precipitated from solution into stacking-fault tetrahedra.[13]

The total concentration of vacant sites $C_V(t)$ was determined from quantitative transmission-electron-microscopy measurements in the following way: After any aging time t, the concentration of vacant sites that have precipitated into stacking-fault tetrahedra is given by $(\frac{1}{4}a)N(t)L^2_{\rm rms}(t)$, where N and $L^2_{\rm rms}$ are the density and mean-squared edge length of the stacking-fault tetrahedra, respectively, and a is the lattice constant.[13] The total concentration of vacant sites remaining in the lattice at any aging time is given by

$$C_V(t) = (\tfrac{1}{4}a)[N(\infty)L^2_{\rm rms}(\infty) - N(t)L^2_{\rm rms}(t)], \qquad (2)$$

where $N(\infty)$ and $L^2_{\rm rms}(\infty)$ are the values of N and $L^2_{\rm rms}$ after the specimen has been fully aged and all of the vacancies have precipitated into stacking-fault tetrahedra.

In order to analyze the positron-lifetime data from the quenched-gold experiments, the data-

analysis techniques described in Ref. 1, which were used for the equilibrium experiments, were extended. This was required by the new configuration of the positron source, as well as by the multiplicity of positron traps in the quenched samples.

The positron source for the experiments on the quenched specimens consisted of ^{22}NaCl sandwiched between two pieces of 6-μm-thick Mylar, which were sealed together with epoxy and an aluminum clamp ring.[11] To allow for positron annihilation in the source, two additional lifetime components were least-squares adjusted to each set of lifetime data. The lifetimes and intensities of the two source components averaged over all the data sets were $\tau_N = 0.43 \pm 0.05$ nsec, $I_N = (21 \pm 1)\%$ which can be associated with annihilations in the sodium chloride and $\tau_M = 1.4 \pm 0.2$ nsec, $I_M = (10 \pm 4)\%$ which can be associated with annihilations in the Mylar sandwich.

The intensity of the annihilations in the sodium chloride is higher than is sometimes observed in positron-annihilation experiments. This is because the NaCl was applied as a sequence of very small drops which were dried onto the Mylar one after another. This resulted in a rather thick coating of salt which, in turn, produced the relatively high percentage of positrons annihilating in the NaCl source material. We are confident that the fitting procedure, which adjusted the lifetimes and intensities of the source components for each data set, could successfully separate these relatively long lifetime components from the much shorter lifetime components which carried the information concerning the trapping of positrons at vacancies. Although the source components could possibly be temperature dependent, this would not have any effect on the results, since the source component was least-squares adjusted in each data set.

In the quenched specimens, there are at least three types of defects which are positron traps: monovacancies, divacancies, and dislocations (primarily those of the stacking-fault tetrahedra). Tests on synthetic data indicated that it would not be possible to separate all three defect components in the data analysis. For this reason, the data were analyzed using a single total trapping rate

$$\kappa(T,t) = \kappa_V(T,t) + \kappa_D(T,t)\,, \tag{3a}$$

where

$$\kappa_V(T,t) = \mu_{1V}(T)\,C_{1V}(t) + \mu_{2V}(T)\,C_{2V}(t) \tag{3b}$$

and

$$\kappa_D(T,\ ) = \mu_D(T)\,C_D(t) \tag{3c}$$

are the trapping rates at vacancies and dislocations, respectively. Here $\mu_{1V}(T)$ and $\mu_{2V}(T)$ are the specific positron trapping rates at monovacancies and divacancies, respectively, at the temperature at which the positron measurements were made, and $C_{1V}(t)$ and $C_{2V}(t)$ are the concentrations of monovacancies and divacancies, respectively, at aging time t. The quantities $\mu_D(T)$ and $C_D(t)$ are the specific trapping rate and concentration, respectively, of atomic sites associated with the stacking-fault tetrahedron dislocations. The latter quantity is calculated from the expression

$$C_D(t) = (3a^2/\sqrt{2})\,L_{\rm rms}(t)\,N(t)\,.$$

Any contributions from other dislocations were taken to be negligible by comparison. Hereafter, the specific variables (T,t) will be omitted in the text for simplicity.

In the least-squares analysis of the positron-lifetime data, the adjustable parameters were the total trapping rate κ; the effective lifetime of trapped positrons τ_V; the lifetime of free positrons in the lattice τ_L; the lifetimes and intensities of the two source components τ_N, I_N, τ_M, I_M; and the parameters associated with the instrumental resolution function.[1] The values of χ^2/ν were typically 1.08 with $\nu = 1500$, indicating a good fit to the data.

The experimental results for the quench-aging experiments are given in Table I. The values of C_V and κ are the best-fit results of the electron-microscopy and positron-lifetime measurements,

TABLE I. Results for quenched specimen Au-1.

Aging time t (h)	$L_{\rm rms}(t)$ (nm)	$10^5\,C_V(t)$	$2C_{2V}/C_V$	$\kappa(T,t)$ (10^9 sec^{-1})	$\kappa_D(T,t)$ (10^9 sec^{-1})	$\kappa_V(T,t)$ (10^9 sec^{-1})
0	0	9.8 ± 0.8	0.75	6.3 ± 0.5	0	6.3 ± 0.5
1.5	25 ± 2	8.9 ± 0.8	0.96	5.6 ± 0.5	0.1 ± 0.1	5.5 ± 0.5
4.0	34 ± 4	7.6 ± 0.9	0.96	3.5 ± 0.6	0.3 ± 0.2	3.2 ± 0.6
6.5	43 ± 3	6.3 ± 1.3	0.96	3.3 ± 0.5	0.3 ± 0.3	3.0 ± 0.6
12.0	47 ± 3	3.7 ± 1.4	0.95	1.1 ± 1.0	0.5 ± 0.5	0.6 ± 1.1
28.0	50 ± 5	1.9 ± 1.4	0.93	1.7 ± 0.5	0.7 ± 0.6	1.1 ± 0.8
∞[a]	55 ± 2	0	...	0.7 ± 0.6	0.7 ± 0.6	0

[a] 60 h at 333 K + 1 h at 408 K.

respectively. The values of κ_D in the table were determined as follows. In the fully aged samples, $\kappa_D(\infty)$ was taken equal to the value of κ deduced from the lifetime measurements. The corresponding value of $C_D(\infty)$ was obtained from the electron-microscopy studies of fully aged samples. From Eq. (3c), the ratio $\kappa_D(\infty)/C_D(\infty)$ yields a value of $\mu_D = (12 \pm 10) \times 10^{13}$ sec^{-1}. This value may be compared with that of 8×10^{13} sec^{-1} found by Cotterill *et al.*[14] for dislocations in quenched aluminum. Within the experimental uncertainties, the two results appear to be consistent. Assuming that μ_D is a constant, and inserting its value into Eq. (3c) together with the measured values of C_D for intermediate aging times yields intermediate values of κ_D. Subtracting these values of κ_D from the measured values of κ gives, according to Eq. (3a), the trapping rate κ_V associated with vacancies only.

Since the electron-microscopy experiments can determine only the total concentration of vacant sites $C_V(t) = C_{1V}(t) + 2C_{2V}(t)$ but cannot separate the relative concentrations of monovacancies and divacancies, it is necessary to make a theoretical estimate of the amount of divacancy formation in the quenched specimens in order to determine values of $\mu_{1V}(T)$. The fraction of vacant lattice sites in divacancies $2C_{2V}(t)/C_V(t)$ was estimated for each aging time by calculating a numerical solution to the time-dependent differential equations which describe divacancy formation,[15] using the value $E_{2V}^B = 0.38$ eV for divacancy binding energy.[16] The calculated values are given in Table I.

We can rewrite the expression for the trapping rate at vacancy defects κ_V in Eq. (3) as

$$\kappa_V = \mu_{1V} C_V (1 - \beta), \tag{4}$$

where

$$\beta = [1 - (\mu_{2V}/2\mu_{1V})](2C_{2V}/C_V).$$

Thus, if we know the ratio $\mu_{2V}/2\mu_{1V}$, we can deduce $\mu_{1V}(t)$ from the experimental results, since all the other quantities are known. Very little is known about the specific positron trapping rate at divacancies μ_{2V}. However, since a single divacancy must be at least as effective at trapping positrons as a single monovacancy, i.e., $\mu_{2V} \geq \mu_{1V}$, then $1 - \mu_{2V}/2\mu_{1V} \leq 0.5$. Thus, taking $\mu_{2V} = \mu_{1V}$ in the expression for β will give an *upper* limit on μ_{1V} at low temperatures, which is required if we wish to establish a *lower* limit on the temperature dependence of this specific trapping rate.

An evaluation of the monovacancy-positron specific trapping rate $\mu_{1V}(T)$ was made by substituting the values in Table I into Eq. (4) and assuming $\mu_{2V} = \mu_{1V}$. A best-fit value of $\mu_{1V}(243\ \text{K}) = (10 \pm 1) \times 10^{13}$ sec^{-1} was obtained. This value is much lower than the value of the specific trapping rate which was deduced from the equilibrium experiments at high

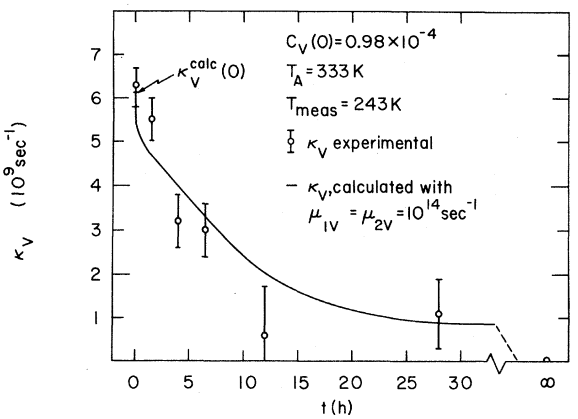

FIG. 2. Rate of positron trapping by vacancies as a function of aging time. Data points were deduced from positron-lifetime measurements, and solid curve is Eq. (4) with $\mu_{2V} = \mu_{1V}$ and $\mu_{1V} = (10 \pm 1) \times 10^{13}$ sec^{-1}. C_V values were taken from Table I.

temperatures, $\mu_{1V}(1033\ \text{K}) = (42 \pm 5) \times 10^{13}$ sec^{-1}.[1] If we assume that this difference is associated with a temperature dependence in $\mu_{1V}(T)$ of the form T^x, we find that the temperature dependence in $\mu_{1V}(T)$ is $T^{1.0 \pm 0.1}$. In Fig. 2 is shown the dependence of the positron trapping rate κ_V upon aging time. The data points were determined from positron-lifetime measurements by subtracting the values of κ_D, as previously explained. The solid curve is the expression for κ_V given in Eq. (4), assuming $\mu_{2V} = \mu_{1V}$, and $\mu_{1V} = (10 \pm 1) \times 10^{13}$ sec^{-1}. The C_V values used are those given in Table I.

III. DISCUSSION

When the above results were first reported,[9] they were found to be inconsistent with measurements of McKee, Stewart, and Jamieson,[7] whose results indicated that the positron trapping rate in their quenched-gold specimens did not change when the temperature was cycled between ice temperature and liquid-nitrogen temperature. Two possible explanations for this discrepancy were considered: The first was that in our quenched specimens the positrons were sampling only a denuded region near the surface (which occurs as a result of vacancy losses during the quench), and that this lowered the apparent trapping rate. The second was that the temperature dependence of the specific trapping rate levels off at low temperatures so that there might be, for example, a large change between 1033 K and ice temperature, and no change between ice temperature and liquid-nitrogen temperature.

To determine whether positrons might have been sampling only a denuded region near the surface rather than the bulk material, comparison measurements were made at 243 K on an electropolished and a nonelectropolished sample cut from a

TABLE II. Comparison of results for quenched-gold specimens Au-1, Au-2, and Au-3.

Specimen Number	Approximate T_{Quench} (K)	Aging time t (h)	Fully Aged L_{rms} (nm)	$10^5 C_V$	$2C_{2V}/C_V$	T_{meas} (K)	$\kappa(T,t)$ (10^9 sec^{-1})	$\mu_{1V}(T_{meas})$ (10^{13} sec^{-1})
Au-2[a]	~1073	1.5	22±2	6.4±0.7	0.96	243	6.7±0.5[a]	20±4
Au-2[b]	~1073	1.5	22±2	6.4±0.7	0.96	243	5.5±0.5[b]	17±3
Au-1	~1133	c	55±2	c	c	243	c,d	12±1
Au-2[a]	~1073	1.5	22±2	6.4±0.7	0.96	298	8.4±1.0[a]	25±5
Au-3	~1073	0	29±2	4.5±1.0	0.64	115	5.5±0.8[d]	18±5
Au-3	~1073	0	29±2	4.5±1.0	0.64	243	7.8±0.8[d]	26±7
Au-3	~1073	0	29±2	4.5±1.0	0.64	298	8.9±1.0[d]	29±8

[a]Electropolished after quench.
[b]Not electropolished.
[c]Specimen Au-1 stepwise aged. See Table I.
[d]Not electropolished, but κ shown has been corrected upward 20%.

a single piece of specimen material which had been quenched from ~1073 K. The electropolishing served to completely remove the region denuded of vacancies during the quench. The results for this experiment on specimen Au-2 are shown in the first and second lines of Table II. It can be seen that the electropolished specimen (first line) has a 20% higher trapping rate than the nonelectropolished specimen (second line). For example, this effect is consistent with values of 10 μm for the positron range and 2 μm for the depth of the denuded zone near the surface. The temperature dependence deduced from the quench-aging experiments on specimen Au-1 should therefore be corrected for this effect by increasing μ_{1V} at 243 K by 20% to $(12\pm1)\times10^{13}$ sec^{-1}. This correction changes the previously deduced temperature dependence from $T^{1.0\pm0.1}$ to $T^{0.9\pm0.1}$. It also has the effect of changing the vertical scale in Fig. 2, but this does not alter the relative agreement between the data and the solid curve. Thus, we can conclude that the idea that the positrons were sampling only a denuded zone near the surface *cannot* account for the positive temperature dependence in the specific trapping rate which we have observed.

We also tested the idea that the temperature dependence levels off at low temperatures. Positron lifetimes were measured at room temperature (298 K) in an electropolished sample from specimen Au-2. The results are shown in the fourth line of Table II. The temperature dependence in μ_{1V}, deduced from the measurements on specimen Au-2, was found to be $T^{1.0\pm1.0}$ between 243 and 298 K. It is of interest to note that in the previous measurements the specimens were cooled to 243 K, and that κ remained constant, indicating that, as expected, the vacancies were frozen in at 243 K. At 298 K, however, κ was found to decay at a rate of 0.5 ± 0.2 day^{-1}, indicating that vacancy annealing was occurring. This made it necessary to extrapolate to zero time the value of κ measured at 298 K. In contrast to this, McKee *et al.*[7] observed no annealing during their measurements which were made at ice temperature. This lack of recovery suggests that vacancies are immobile at ice temperature or that these authors may have been measuring effects arising either from dislocations introduced during their quench, or from vacancy precipitates in their relatively impure material.[7,13]

In order to further investigate the temperature dependence in μ_{1V} at low temperatures, positron lifetimes were measured at 115, 243, and 298 K in a third specimen Au-3 quenched from ~1073 K. The results of these measurements are shown in the last three lines of Table II. The temperature dependence of μ_{1V} between 115 and 298 K for this specimen is well described by $T^{0.5\pm0.3}$.

The deduced values of μ_{1V} as a function of temperature for the various specimens are plotted in Fig. 3. We see that although there is consistently a positive temperature dependence either when quenched specimen results are compared with the equilibrium results or when results for each specimen are considered individually, there is a vari-

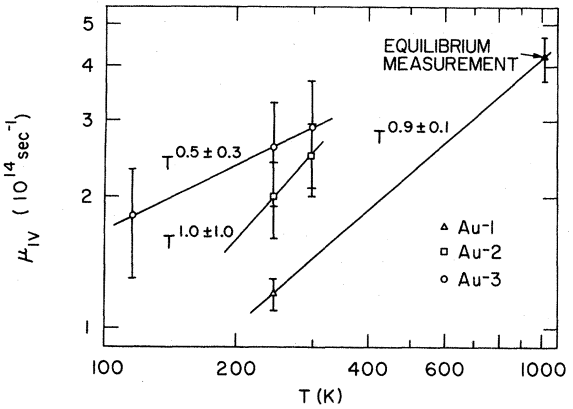

FIG. 3. Specific trapping rate as a function of temperature.

TABLE III. Lifetime in the lattice τ_L and lifetime in the vacancy trap τ_V for various temperatures T.

T (K)	τ_L (10^{-12} sec)	τ_V (10^{-12} sec)
115	95 ± 5	220 ± 20
243	110 ± 8	210 ± 6
673–1033	121 ± 2	210 ± 2

ation among the quenched specimens in the absolute values of $\mu_{1V}(T)$ obtained. This variation can be explained in the following manner. It has been reported that the vacancy concentration deduced from stacking-fault tetrahedra can be underestimated when the tetrahedra are small (~30 nm).[17] Since the rms size of the tetrahedra in the various states of specimen Au-1 was generally greater than 30 nm, while for specimens Au-2 and Au-3 $L_{\rm rms}$ was 22 and 29 nm, respectively, it is probable that the absolute vacancy conentrations for Au-2 and Au-3 were underestimated. No such problem is likely to have occurred with specimen Au-1. Furthermore, the value of μ_{1V} (243 K) deduced from Au-1 resulted from six independent sets of measurements of κ and C_V. For these reasons, the absolute value of μ_{1V} determined from specimen Au-1 is considered to be more accurate than those from specimens Au-2 and Au-3. Nevertheless, the results obtained for specimens Au-2 and Au-3 individually concerning the positron range and the temperature dependence of μ_{1V} at low temperatures are still valid, since only relative values of κ and C_V were required. It is also important to point out that the microscopy of these specimens provides a *lower* limit on C_V, and hence a *lower* limit on the temperature dependence of μ_{1V} obtained by comparison with the equilibrium results.

We wish to make a final comment concerning the temperature dependence of the positron lifetime in the lattice τ_L and the lifetime in the vacancy trap τ_V. In the least-squares analysis of the positron-lifetime data, the values of τ_L and τ_V were adjusted independently for each temperature. The resulting values for τ_L and τ_V as a function of temperature are shown in Table III. The lifetime in the vacancy trap appears to be temperature independent within the uncertainties, but the lifetime in the lattice appears to have a slight temperature dependence of $(3\pm 1)\times 10^{-14}$ sec K^{-1}. This represents a fractional change in the lifetime of $(3\pm 1)\times 10^{-4}$ K^{-1}, which is about seven times the coefficient of volume expansion, thus precluding a simple correspondence between lifetime change and volume change.

IV. CONCLUSIONS

We find that the equilibrium results alone indicate a temperature dependence of μ_{1V} of $T^{0.5\pm 0.2}$ over the range from 673 to 1033 K. The results from a comparison of the most accurately characterized quenched specimen Au-1 with the equilibrium measurements indicate a temperature dependence of $T^{0.9\pm 0.1}$ between 243 and 1033 K. The results of measurements over the low-temperature range from 115 to 298 K consistently gave a position temperature dependence. Thus, the results of the equilibrium experiments and the quenching experiments are in agreement and together strongly indicate a positive temperature dependence in the positron specific trapping rate over the entire temperature range from 115 to 1033 K.

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