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**POSITRON PREVAANCY EFFECTS IN PURE ANNEALED METALS\***

Lars C. Smedskjaer  
Materials Science Division  
Argonne National Laboratory  
Argonne, Illinois 60439 U.S.A.

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**MASTER**

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Lars C. Smedskjaer  
Materials Science Division  
Argonne National Laboratory  
Argonne, Illinois 60439 U.S.A.

### SUMMARY

The low-temperature prevacancy effects sometimes observed with positrons in well-annealed high-purity metals are discussed. It is shown that these effects are not experimental artifacts, but are due to trapping of the positrons. It is suggested that dislocations are responsible for these trapping effects.

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## 1. Introduction

During the last decade, the utilization of the positron as a probe in metals has increased dramatically. The positron is commonly used both to study the electronic structure in perfect materials and to probe for lattice imperfections.

Following the early observations of the angular correlation of the annihilation radiation (1), the first experiments aimed at measuring the electron momentum density in supposedly perfect metals were done (2-4). While these experiments were concerned with the electronic structure in perfect metals, the potential of the positron as a probe for lattice imperfections was not fully realized until ~ 1968-1970. The first observations of positron trapping (5-9) became important through their subsequent interpretation by the trapping model (10-12), which describes positron behavior in terms of a radioactive decay scheme.

While the early experiments provided ample evidence for positron trapping in vacancies (8,9), later experiments provided evidence for positron trapping in dislocations (13) and voids (14) as well. Thus, the positron was shown to interact strongly with lattice imperfections containing a region of low electron density. This interaction between the positron and lattice defects was and still is an unwelcome complication for those interested in the electronic structure of perfect metals. On the other hand, this same interaction offers a valuable research tool for those of us interested in defect physics.

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The prevacancy effects, which are the subject of this lecture, might also at first be seen as undesirable. However, as we shall see, the prevacancy effects are due to positron interactions with defects and should therefore not be thought of as a complication introduced by the positron but rather as reflecting physical properties of lattice defects in metals.

The aim of the present lecture is first to establish that the prevacancy effects are real and not experimental artifacts. Next we will establish that the prevacancy effects are extrinsic in nature and not characteristic of the perfect material. We will then show that the prevacancy effects are caused by positron trapping in defects, and finally, suggest the possibility that the defects in question are dislocations.

## 2. Evidence for Prevacancy Effects

For positron annihilation in a perfect metal the positron lineshape parameter and lifetime are expected to behave as almost linear functions of temperature except at the lowest temperatures (15,16). An experimental situation close to that of a perfect metal may be realized with a high-purity, well-annealed sample at temperatures below those where thermally generated vacancies can cause significant positron trapping. In this low-temperature region, referred to as the prevacancy region, the prevacancy effects manifest themselves as significant deviations from the expected linear temperature dependence of the positron signal.

Although the prevacancy effects at present have only been experimentally observed at low temperatures, we have no a priori knowledge with regard to their high-temperature properties; thus for example, we do not know whether their presence will affect the vacancy formation enthalpy measurements performed using positron annihilation. Later in this lecture, it will be suggested that such high-temperature effects are unlikely if the sample has a low dislocation density.

Since the prevacancy effects were first observed for supposedly well-annealed, high-purity samples (17,18), and have since been consistently observed in such samples, the present considerations will also be limited to high-purity, well-annealed samples.

In Fig. 1 three schematic examples of prevacancy effects are shown along with the expected behavior of the positron signal for a perfect metal. Fig. 2a illustrates an experimental example of a prevacancy effect for Ta (19,20). The behavior in Fig. 2a corresponds to the behavior represented in Fig. 1b, hereafter designated "type 1-b behavior". For comparison, Fig. 2b shows an example of a behavior presumably more closely related to the expected behavior of a perfect metal (21).

The metal Cd, despite its lattice structure and anisotropy, has attracted the most interest with respect to prevacancy effects. This may be, in part, because the prevacancy effects in this metal are commonly large, although large prevacancy effects have been observed in other metals as well. The first observations for Cd (17,18) were made with Doppler-broadening, but these were rapidly followed by both angular-correlation (22) and lifetime (23) experiments. These observations were all very similar to the behavior depicted in Fig. 1a, which may, however, sometimes be difficult to separate experimentally from type-1b behavior. Further evidence for the prevacancy effect was obtained in polycrystalline In, Zn, Cd, Au, and Pb (24,25,26) where the Doppler-broadening results showed behavior similar to that of Fig. 1a. However, the lifetime results (26) for Au and Cd seemed to be more closely related to the behavior depicted in Fig. 1c. Very pronounced type-1b behavior has been found for In (27), as has a much less pronounced behavior in Fe (20). Recent results for Cd single crystals also exhibited a shoulder effect as in Fig. 1b (28). For Ni, a variety of behaviors have been

observed. A transition from the flat region to the steeper region was found  $\sim 250$  K (29), as was also the case for Fe and V (29) at slightly different temperatures. Other work on Ni suggests a slope change at  $\sim 700$  K (30) and  $\sim 900$  K (31). Lifetime results (32) also indicated a prevacancy effect for Al, Ag, and Au.

To summarize, at present we have ample evidence for prevacancy effects in many metals: Al (32), Ag (32), Au (24,26,32), Pb (25), In (24,27), Zn (24), Ni (29-31), Fe (20,29), V (29), Ta (19,20), and Cd (17,18,22,23). No effort has been made here to mention every experiment showing a prevacancy effect; the above list serves only to emphasize that prevacancy effects have been observed by all three positron techniques, for so many metals and by so many different groups that it is safe to assume that the prevacancy effects are not experimental artifacts related to one particular group, technique or metal.

### 3. The Extrinsic Nature of the Effects

We shall now consider whether the effects are characteristic of the perfect material (intrinsic) or whether they are extrinsic.

It is noteworthy in regard to the prevacancy effects (Fig. 1) that no hysteresis with respect to the temperature dependence of the positron annihilation parameters has been reported, except after high-temperature annealing (43). This could at first be regarded as evidence that the effects are intrinsic. However such a conclusion would be premature, since the type-1b temperature dependence for In observed in ref. (27) was not reproduced by other investigators (33,24). Correspondingly, a type-1b temperature dependence observed for Ta (19,20) was not reproduced in a later measurement by the same group (21) (see Fig. 2a, b). It is interesting to note that the type-1b behavior has now also been found for single-crystal Cd (28,34). A comparison among lifetime results (35,26,32) for Au has been made in

ref. (35): good agreement for  $T \gtrsim 300$  K was found, while substantial disagreement was present at lower temperatures. In the same work (35), a normalized comparison between the Doppler-broadening data from (35) and (26) was also made and it was found that the low-temperature data deviated substantially from one another. As mentioned previously, a broad variety of effects have been observed in Ni. The lifetime results of ref. (31) showed a temperature independent region below  $\sim 900$  K followed by a steeper temperature dependence until the vacancy onset at  $\sim 1100$  K. No corresponding effect is found in Doppler-broadening (36-38) or in peak-count (39,30) measurements. The peak-count measurements of ref. (30) showed a transition at  $\sim 700$  K. No corresponding effect was reported by (36-39). Below room temperature the Ni data are scarce, but it might be noted that the transition around  $\sim 250$  K (29) is not visible in the data of ref. (38).

Based upon the present comparison for Ni, Ta, In, and Au, we are led to conclude that the prevacancy effects are extrinsic, at least for these metals. Furthermore, lifetime results for Cd (40) show that the positron annihilation spectrum contains at least two lifetimes in the temperature region where a prevacancy effect is observed. Thus, one concludes that the prevacancy effects for Cd are also extrinsic and it seems reasonable to assume that this conclusion is valid for other metals as well.

#### 4. Interpretation of the Prevacancy Effects

In the original work demonstrating prevacancy effects (17,18), it was suggested that the positron was trapped by transient dilatations in the perfect lattice. In another interpretation, the concept of self-trapping (41) was introduced. According to this suggestion, the positron was able to "dig" a hole in the lattice and subsequently become trapped. Both of these interpretations as well as a more recent suggestion (42), consider the

prevacancy effects to be of an intrinsic nature. The present observation that prevacancy effects are extrinsic precludes these suggestions with respect to the prevacancy effects discussed here.

The present interpretation of the prevacancy effects follows that given in ref. (35), where it was suggested that the simple straight-line behavior observed in that study for the lineshape and lifetime in Au over the range  $27 \text{ K} < T < 592 \text{ K}$  might be close to the intrinsic behavior, while the longer lifetimes reported by others (26,32) for  $T < 300 \text{ K}$  could be caused by positron trapping. This view of the prevacancy effects as the result of positron trapping is further supported by the observation of positron trapping at room temperature in supposedly well-annealed Cu (43). In the latter work, it was further demonstrated that the amount of trapping at room temperature depends upon the mechanical history of the sample prior to the anneal. This work is of particular value because the possibility of artifactual differences between results from different laboratories, obtained with different samples and equipment, is eliminated.

Further support of the present viewpoint is obtained from the lifetime results for Cd (40). For a Cd sample with a mean-lifetime temperature dependence (see Fig. 3) similar to that depicted in Fig. 1a, a second long lifetime was resolvable for temperatures below  $\sim 150 \text{ K}$ . The intensity of this second lifetime as a function of temperature is shown in Fig. 4. The existence of two lifetimes is direct evidence for positron trapping.

Thus, in conclusion, the prevacancy effects in Au, Cd and Cu are due to positron trapping in defects. One may suggest that this would be the case for other metals as well.

## 5. The Nature of the Defects

Nothing conclusive is known at present about the defects causing the prevacancy effects. One may suggest high-angle grain boundaries, dislocations, or impurity-related defects (e.g., pinned dislocations), or any combinations of these, as possible causes. Without precluding that any of these defects could cause or contribute to a prevacancy effect, one might note that grain boundaries alone are unlikely to have caused the presently observed effects, since grain sizes in annealed metals are commonly large. In particular, one might note the recent observations of prevacancy effects in Cd single crystals (24,28,34).

The experiments on Cu (43) and Cd (40) do, however, provide some important clues. First, both experiments showed that the lineshape (Cu) or lifetime (Cd) characteristic of the defect was similar to that of a vacancy. This may be interpreted to mean that the binding energy between the defect and the positron is high ( $> 1$  eV). Second, the intensity of the long lifetime in Cd (see Fig. 4) was shown to decrease with increasing temperature below 300 K, while the increase above 300 K is due to positron trapping in thermally generated vacancies. The decrease in intensity corresponds to a decrease in the specific "effective" positron trapping cross section. This could be caused either by diffusion-limited trapping or by a low binding energy ( $< 0.1$  eV) between the positron and the defect.

Without ruling out the possibilities of diffusion-limited trapping we shall, following the work of ref. (44), explore the contrast between the binding energies. According to the suggestions made in ref. (44), the dislocation is a positron trap containing two localized positron states, one with a high binding energy and one with a low binding energy. The dislocation line itself is considered to have a low positron binding energy, while, for

example, jogs on the dislocation lines have a high positron binding energy. It was shown that the positron may at first be trapped on the dislocation line emitting a phonon in the process. Once trapped on the line, the positron probes the line and may in the process either annihilate, detrap, if it collides with a phonon, or get further trapped by a jog. If the positron annihilates while probing the line, it will result in a lineshape or lifetime close to that of the perfect material, but if it annihilates while trapped in a jog, the annihilation characteristics resemble those of a vacancy. The predictions of this model for the mean positron lifetime in Al, assuming a temperature-independent jog density, is shown in Fig. 5 for various binding energies and jog densities. For convenience, the jog density has been expressed in terms of the transition rate between the dislocation-line trapped state and that of the jog. For comparison, the expected behavior of the mean lifetime for positrons in the perfect lattice is also shown. From Fig. 5, it is seen that prevacancy effects of the types depicted in Fig. 1a and Fig. 1c can be explained by this model.

The shoulder-like effect characteristic of type-1b behavior appears just below the vacancy region. At such high temperatures, the assumption of a temperature-independent jog density cannot be justified, and the results of ref. (44) should be correspondingly revised. The effects of a temperature-dependent jog density are easily investigated (45) for the simple case where the temperature-independent jog density is low. It was found that a shoulder effect could develop when the activation enthalpy for jog formation was less than that for vacancy formation, which is very likely, and that the magnitude of the effect would be proportional to the dislocation density.

Thus, the model suggested by ref. (44) is qualitatively consistent with the experimental observations of the prevacancy effects, although it

represents an oversimplification of the real physical situation at hand. For a real sample, one anticipates that not only one but several dislocation types would be present, which implies that a spectrum of positron dislocation-line binding energies, rather than a single energy, should be considered. One may, however, speculate that the prevacancy effects might yield information with respect to dislocation-line properties such as jog densities and activation enthalpies for jog formation.

The present suggestion that prevacancy effects can be caused by positron trapping in dislocations does not preclude other explanations. It should be emphasized that other two-trap systems similar to that proposed for the dislocation could result in similar temperature effects as well (e.g., dislocation networks).

Finally, attention must be drawn to the possibility of diffusion-limited positron trapping. It is pointed out in ref. (46) that positron trapping in dislocations would be strongly temperature-dependent if the trapping process were diffusion-limited and it is possible that this description could account for the prevacancy effects as well.

In conclusion, we have suggested that the prevacancy effect can be due to positron trapping in dislocations and we have shown that the model proposed in ref. (44) can account qualitatively for the observed results. Clearly, experimental work on systems containing precisely characterized dislocations will be needed in order to provide a final conclusion with respect to the present suggestions.

## 6. The Prevacancy Effects and Other Measurements

In the present section we shall briefly consider how the prevacancy effects may interfere with positron experiments.

It was stated in the Introduction that the positron is sensitive to regions of low electron density. The prevacancy effects may therefore interfere with most other positron experiments (e.g., low temperature annealing experiments and vacancy formation enthalpy measurements) to the degree that low-density regions other than those of "interest" are present in the sample. This complication is due not to the positron behavior, but to defect physics itself, although the present author finds it deplorable that the positrons have not read a standard textbook on dislocations and vacancies and are thus unable to distinguish clearly between them.

The irreproducible nature of the prevacancy effects also indicates that the interference may be avoided or reduced by sufficient in situ annealing. The proposed model of ref. (44) strongly suggests that if the transition between the flat and the steeper part of the temperature dependence of the positron signal (see Fig. 1) takes place at a sufficiently low temperature, then parameters such as vacancy formation enthalpies can be correctly determined by means of positron annihilation; however, extreme caution should be exercised with respect to such determinations if, for example, type-1b behavior is seen. Low-temperature annealing experiments may also be affected when weakly-binding dislocations are present. Possible changes in both type and density of such dislocations upon annealing will make it difficult to separate dislocation effects from the effects of interest. Thus, in experiments where the prevacancy effects are not of primary interest attempts should be made to avoid them.

## 8. Conclusion

It has been shown that the prevacancy effects observed for positron annihilation in metals are due to positron trapping in defects. It has been suggested that these defects are dislocations, but other possibilities should also be considered in future work.

One might at first consider the prevacancy effects as a complication, just as the discovery of positron trapping in defects might have been perceived originally. However, since these effects reflect the underlying metal physics, one might speculate that the prevacancy effects may represent a future area of defect research.

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## Figure Captions

- Fig. 1. Three schematic examples of prevacancy effects. The dashed curves show the expected intrinsic behavior of the positron annihilation parameter  $F$  (mean lifetime or lineshape) vs temperature  $T$  at low temperatures. The solid curves show the experimentally observed temperature dependence when prevacancy effects are present.
- Fig. 2. Wing parameter,  $W$ , as a function of temperature for Ta, (a) with and (b) without apparent prevacancy effect. From ref. (19-21).
- Fig. 3. Mean positron lifetime  $\bar{\tau}$  as a function of temperature for Cd. Positron trapping in thermally generated vacancies is apparent in the two high-temperature points. The high-temperature part of the curve is not shown (compare with Fig. 1a). From ref. (40).
- Fig. 4. Intensity  $I_2$  as a function of temperature for Cd sample with prevacancy effect. Trapping in thermally generated vacancies is seen above 300 K. Trapping in defects (prevacancy effect) is visible below 150 K. From ref. (40).
- Fig. 5. (a-e) Calculated mean positron lifetime in Al for a dislocation density of  $\sim 10^8 \text{ cm}^{-2}$  and the following values of positron dislocation-line binding energy ( $E_b$ ) and positron trapping rates into jogs ( $\zeta$ ): (a)  $E_b = 0.1 \text{ eV}$ ,  $\zeta = 10^3 \lambda_b$ ; (b)  $0.1 \text{ eV}$ ,  $10^2 \lambda_b$ ; (c)  $0.03 \text{ eV}$ ,  $10^3 \lambda_b$ ; (d)  $0.03 \text{ eV}$ ,  $10^2 \lambda_b$ ; (e)  $0.01 \text{ eV}$ ,  $10^3 \lambda_b$ . The quantity  $\lambda_b$  is the annihilation rate in the perfect material at  $T = 0 \text{ K}$ ; (f) Expected mean positron lifetime in a perfect lattice. From ref. (44).

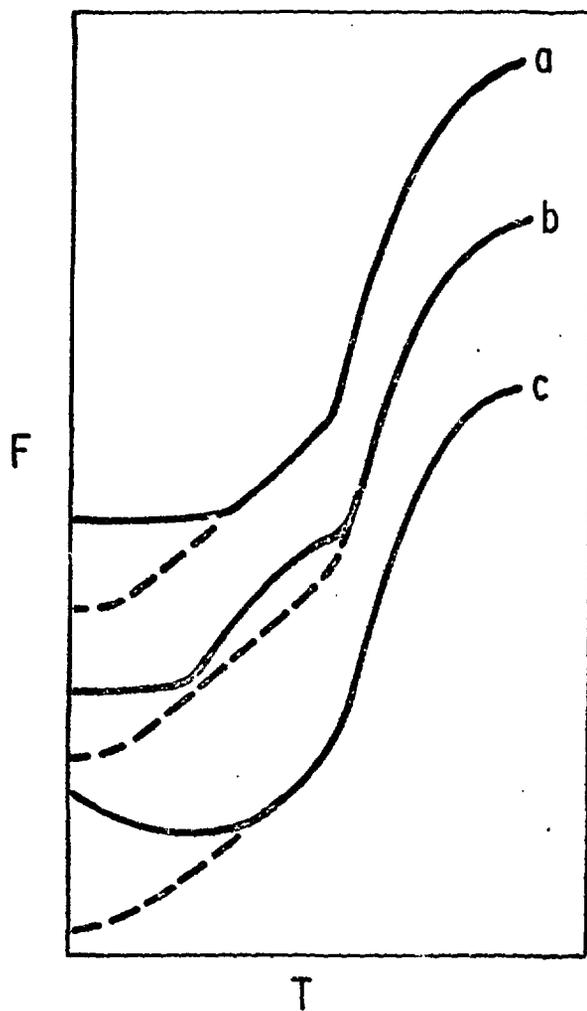


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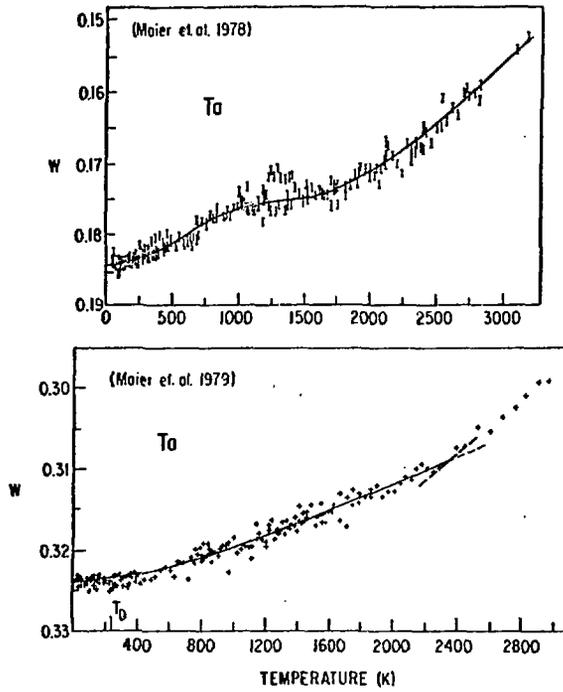


Fig. 2. Wing parameter,  $W$ , as a function of temperature for Ta, (a) with and (b) without apparent prevacancy effect. From ref. (19-21).

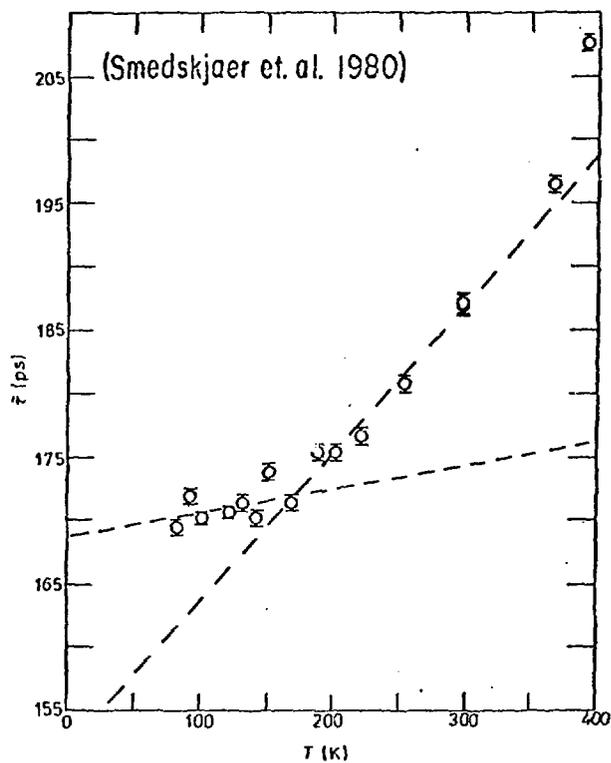


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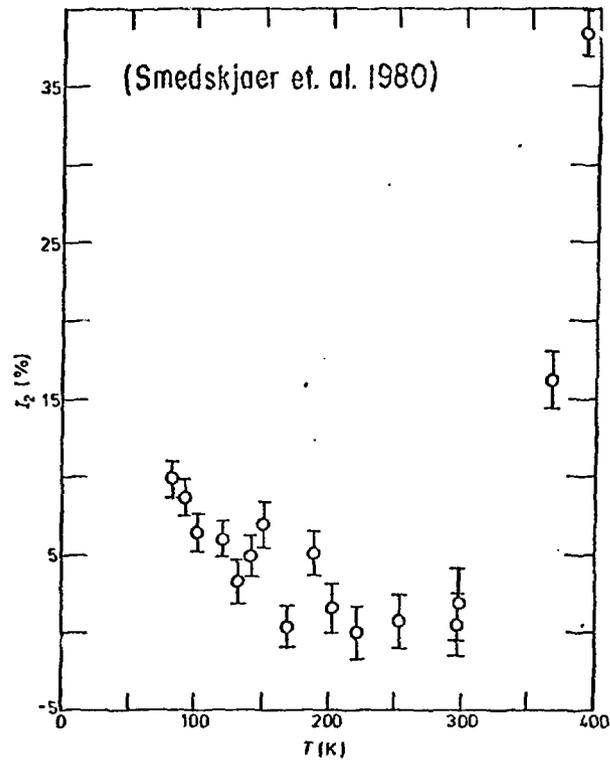


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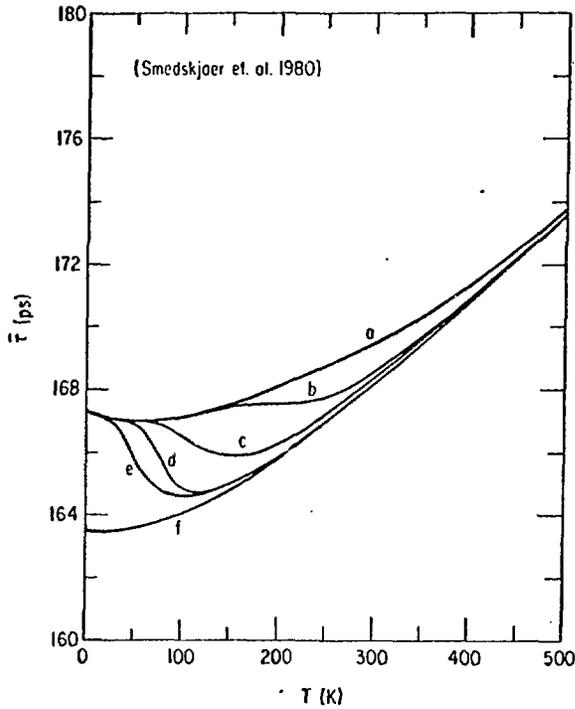


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