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POSITRONS IN AMORPHOUS ALLOYS

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ABSTRACT

Positron annihilation techniques give interesting informations about "empty spaces" in amorphous alloys.

The results of an extensive research work on the properties of either pre-existing or irradiation induced "empty spaces" in four amorphous alloys are presented. The pre-existing empty spaces appear to be small vacancy-like defects. The irradiation induced defects are "close pairs" with widely distributed configurations. There is a strong interaction between vacancy like and interstitial like components.

A model is proposed, which explains the radiation resistance mechanism of the amorphous alloys.

INTRODUCTION

This paper describes recent progress made in the field of amorphous metals (metallic glasses) using the positron annihilation techniques.

In the first part, some details about structure, preparation and properties of amorphous alloys are given. A brief review introduces the interesting open questions raised by previous works.

In the second part, an extensive joint research work to study four amorphous alloys, $Fe_{80}B_{20}$, $Fe_{40}Ni_{40}P_{14}B_6$, $Cu_{50}Ti_{50}$ a,d $Pd_{80}Si_{20}$ is summarized. In this work [1-3] the authors try to answer the remaining open questions about empty spaces and radiation induced defects in the amorphous alloys considered.

1. Overview of amorphous alloy properties [4]

1.1. Structure of amorphous alloys [5]

The essential feature of an amorphous metal is the lack of long range atom periodicity. Most of the amorphous alloys show an atomic packing fraction between 0.66 and 0.68. This is smaller than the corresponding value for f.c.c. or h.c.p. packing (0.74) but similar to the value for the b.c.c. structure (0.68) [6].

Several structural models have been proposed. In the microcrystallite models, the structure is made of an assembly of uncorrelated crystalline regions, the size of which is some 10 Å. To explain experimental results, local strain, packing faults and dislocations were introduced [7-8]. However some difficulties remained and continuous random models were developped.

The Dense Random Packing of Hard Spheres (DRPHS) models were derived from the Bernal model made in order to explain the structure of simple liquids [9]. A review of these models for amorphous alloys is given in [10]. Such a structure presents different kinds of empty spaces : the BERNAL HOLES. Cargill made an evaluation of the canonical hole parameters in a dense random packing of Fe atoms [6]. Some examples of empty space type and volume are : tetrahedron : 0.01 ; octahedron : 0.09 ; dodecahedron : 0.19 ; trig. prism : 0.31 ; archimedian antiprism 0.38 (the unit is the free volume of the single vacancy in crystalline Fe). Such DRPHS models are in good agreement with experimental results for most metallic glasses.

1'2. Amorphous alloy preparation [11]

The amorphous structure is obtained by a rapid quenching of the liquid metal or alloy (cooling rate 10^6 K s^{-1}) which freezes the liquid structure.

Experimentally, in most of the pure metals, the amorphous structure is stable only below room temperature. Improved stability is obtained in the case of metal-metalloïd alloys by filling most of the Bernal holes with atoms like P, B, Si or C. Considering metal-metal alloys, the amorphous state is obtained by quenching markedly diffe-

ferent sized constituent atoms with deep eutectic composition.

The most widely used technique is the melt spinning : the molten metal is spread in a thin layer on a rapidly rotating cylinder; ribbons of 20-50 μm are obtained with a production rate of 30 m s^{-1} [12-14].

1'3. Interest of the amorphous alloys [15]

Properties of amorphous alloys differ strongly from those of the corresponding crystalline alloys. A significant part of the research work in this field is justified by potential applications of these materials. These alloys appear to be interesting in several domains.

- Magnetism : a soft ferromagnetism is presented by Fe, Ni, Co based amorphous alloys. Very high permeability and low energy losses can be achieved by thermal treatment at temperatures below crystallization [16-19].
- Mechanical properties : the highest Ultimate Tensile Strength is obtained with $Fe_{80}B_{20}$ alloy (370 kg mm^{-2}) [20]. The highest specific UTS is presented by $Ti_{50}Be_{40}Zr_{10}$ (UTS = 240 kg mm^{-2} ; density = 4.1 g cm^{-3}).
- Nuclear Energy : the amorphous alloys seem to present a good resistance to radiation damage [21-22].
- Hydrogen Storage : the storage capacity of some metallic glasses [15] appears to be higher than that of the corresponding crystalline alloy.

1'4. Previous work on amorphous alloys using positron techniques

In crystalline metals, positron annihilation techniques have enabled great progress to be made in understanding the nature of point

defects. Positrons detect vacancy type defects even at concentrations of a few ppm. The lifetime value of trapped positrons gives information on the size of submicroscopic vacancy agglomerates and microvoids.

In amorphous alloys, positron studies appeared of great interest as soon as a surprisingly high lifetime was detected in as received amorphous specimens [21-24]. One of the question was whether this high lifetime corresponds to a delocalized state of the positron or to a trapped state in the empty Bernal holes [25].

Other questions arose because the positron annihilation characteristics appeared to be only weakly influenced by the physical state of the alloys. Thermal treatments at temperatures below crystallization cause almost no change in the positron parameters [26-27]. Attempts to create defects by room temperature electron irradiation [21-22] or room temperature cold work [28-29] were unsuccessful. Crystallization seems to reduce the positron lifetime values only slightly [27].

At that point, a cooperative work was initiated to study the remaining open question about pre-existing empty spaces and irradiation induced defects. This work is summarized below.

2. Positron studies in $Fe_{80}B_{20}$, $Fe_{40}Ni_{40}P_{14}B_6$, $Cu_{50}Ti_{50}$ and $Pd_{80}Si_{20}$

"Irradiation induced holes" are studied in the four alloys after 20 K electron irradiation. They are also investigated in $Fe_{80}B_{20}$ after 28 K neutron irradiation. Although "pre-existing empty spaces" have previously been investigated, some classical experiments on the as-received or as-annealed state are performed in $Fe_{80}B_{20}$ and $Fe_{40}Ni_{40}P_{14}B_6$ in order to obtain a reference.

Information about the size of "empty spaces" and "irradiation induced holes" are obtained by positron lifetime measurements. Their mobility is determined in $Fe_{80}B_{20}$ and $Fe_{40}Ni_{40}P_{14}B_6$ by the magnetic relaxation technique [30].

2'1. Experimental aspect

Specimens are prepared by melt spinning : $Fe_{80}B_{20}$ CEN-Grenoble (F. Vanoni); $Cu_{50}Ti_{50}$, $Pd_{80}Si_{20}$ Basel University (H. Kunzi); $Fe_{40}Ni_{40}P_{14}B_6$, Allied Chemical Corporation.

Electron irradiations are performed with a 3 MeV Van de Graaff. The specimens are immersed in a liquid hydrogen cryostat in line with the accelerator. Neutron irradiations are carried out in a 28 K liquid neon loop. In both cases, specimens are transferred into the positron lifetime device at 77 K. A sandwich-type ^{22}Na source-sample arrangement is prepared at liquid nitrogen temperature.

The lifetime spectrometer is a fast slow coïncidence ORTEC system [31] (the ^{207}Bi decay constant measured in experimental conditions is 187 ps). After irradiation, positron lifetime spectra are measured at 77 K at each step of an isochronal thermal treatment, Δt = 30 min, and ΔT = 25 or 50 K. In all other experiments, spectra are also obtained at 77 K, except where specified.

2'2. Experimental results

After source and background corrections all spectra of amorphous alloys are well analyzed with one single exponential decay component. Attempts to fit two lifetimes are always unsuccessful.

2'2.1. Lifetime measurements in as received alloys

Table I gives positron lifetime measured at 77 K for the four

alloys in as-received state. For the sake of comparison the table also gives lifetime values in the constituent metallic elements of the alloys, either well annealed or irradiated up to positron trapping saturation.

2'2.2. Effect of the measurement temperature

The lifetime of the as received-state $Fe_{80}B_{20}$ measured at 300 K, 77 K and 4 K are respectively 144 ± 1 ps, 142 ± 1 ps and 138 ± 1 ps. A significant decrease is observed between 300 K and 4 K.

Kajcos et al [32-33] and Kögel [25] found a temperature dependent line shape parameter for Doppler broadening in $Fe_{80}B_{20}$ and other METGLAS [34] alloys (however the line shape parameter of Doppler broadening for the amorphous alloys remained constant below 77 K).

2'2.3. Effect of thermal treatment

As received $Fe_{80}B_{20}$ and $Fe_{40}Ni_{40}P_{14}B_6$ amorphous specimens are heated linearly from room temperature to 500°C. A deep, broad magnetic relaxation zone is found between 100 and 300°C as previously observed [35-37].

Another set of similar specimens is isochronally annealed (Δt = 1 hour ; ΔT = 100°C ; T_{max} 500°C). Initial magnetic permeability and positron lifetime are measured simultaneously.

Annealing below the crystallization temperature (Tc ≈ 400°C) induces, as described in previous experiments [16-19, 26, 38] an increase of the initial magnetic permeability and a very weak decrease of the lifetime. In $Fe_{40}Ni_{40}P_{14}B_6$, the initial permeability is increased by a factor of five and the positron lifetime is decreased of 2 ps after an annealing of 2 hours at 350°C.

Annealing at temperatures just above the crystallization point induces a lifetime decrease of 5 ps in the four alloys, but the lifetime level is still well above the values measured in carefully annealed pure metals. In $Fe_{80}B_{20}$ annealed under 10^{-11} bar at 800°C for two hours, positron lifetime is only 120 ps. This value is not far from those observed in annealed Fe (110 ps) measured with the same lifetime spectrometer.

Preliminary experiments are made to test effects of a variation in the cooling rate*. $Fe_{81,5}$ $B_{14,5}$ Si_4 specimens of different thicknesses are quenched from the liquid alloy with different cooling rates. However, all specimens have the same positron lifetime (within one ps!). These results are now checked for the four alloys studied.

2'2.4. Hydrogenation [39]

The amorphous alloy $Pd_{80}Si_{20}$ presents a good hydrogen storage capacity [15]. One explanation is given in terms of hydrogen location in the voids existing in the amorphous structure [40-41].

Positron lifetime is measured in $Pd_{80}Si_{20}$ before and after hydrogen charging. There is no significant difference between the positron lifetime observed in the hydrogenated or deshydrogenated state (respectively 154±1ps and 155±1ps). In order to introduce hydrogen into the specimens, they are heated for 2 hours at 100°C under 1 bar of hydrogen. Hydrogen dissolution is checked by electrical resistivity measurements (increase of 1 %). The charging process

*Specimens kindly prepared by Prof. F.E. Luborsky

is reversible and can be repeated several times after hydrogen evacuation under 10^{-8} bar , at room temperature.

2'2.5. Irradiation effects

2'2.5.1. Electron irradiation

Table I gives in column 3 positron lifetimes measured in the four amorphous alloys after a 20 K, 1.4 x 10^{19} e^{-} cm^{-2} electron irradiation followed by transfer at 77 K. A significant increase of the positron lifetime is induced by the low-temperature irradiation. In pure metals, such a dose induces a positron trapping saturation state and the observed values are thus equal to the positron lifetime trapped at a single vacancy. These last values are given in column 4 for the metallic components of the alloys. Comparison of columns 3 and 4 shows that positron lifetimes in the irradiated amorphous alloys are slightly lower than the vacancy value in the alloys components.

The effect of the electron irradiation dose on positron lifetime in amorphous $Fe_{80}B_{20}$ appears on Fig. 1. At any dose, the spectra are described well by one single component. As a function of the electron dose, the positron lifetime increases continuously till a limit value close to the vacancy positron lifetime in iron.

The positron lifetime recovery of the electron irradiated specimens as a function of the annealing temperature is presented on Fig. 2: the lifetime decreases continuously. After heat treatments around room temperature, the positron lifetime values in the as-received states are recovered.

2'2.5.2. Neutron irradiation [42]

When a boron alloy is irradiated in a nuclear reactor, a nuclear reaction $^{10}B(n, \alpha)$ occurs and the emitted particles (α : 1.8 MeV ; ^{7}Li : 1 MeV) create a significant radiation damage [43].

Amorphous $Fe_{80}B_{20}$ specimens are irradiated with various doses. Positron lifetime measured at 77 K increases and reaches saturation for doses around 0.5 10^{17} n v t > 1 MeV. Surprisingly, the saturation limit value is about the same as the saturation positron lifetime value obtained after electron irradiation. Moreover, the positron lifetime recovery curve fits exactly with that observed after electron irradiation as shown in Fig. 2.

2'2.5.3. Magnetic relaxation after electron irradiation [1]

$Fe_{80}B_{20}$ and $Fe_{40}Ni_{40}P_{14}B_{6}$ alloys are studied by magnetic relaxation techniques [30] after 20 K electron irradiation and transfer at 77 K. The following results are obtained :

- the continuous recovery of the irradiation defects is confirmed between 77 and 300 K,
- at each annealing temperature , a constant fraction of the radiation induced magnetic relaxation anneals out ,
- this annealing occurs after very few atomic jumps (less than 6),
- there is no evidence that irradiation induces any defect performing a long range migration.

2'3. Discussion

2'3.1. Preexisting "empty space"

In as received alloys, a high positron lifetime is observed. The

question is whether it corresponds to a delocalized or localized state of the positron. A convincing answer is proposed by R. Kiemnen [44] :

"In the amorphous to crystalline transition, there is only a small change in the mean atomic and electronic density (of the order of 1 %). It is therefore not very plausible to assign the lifetime increase in the amorphous phase to disorder induced effects in a delocalized positron state, which mainly produces smearing in the momentum distribution. Trapping into open spaces in the amorphous structure would seem the most natural explanation".

Additional evidence of positron trapping in as received amorphous alloys is given by the positron lifetime temperature dependence (§ 2°2.2.). Although the overall lifetime change is only 6-8 ps, it is certainly more than that expected on the basis of a small thermal expansion [45] . This suggests some weakly temperature dependent mechanism for the positron trapping or localization in amorphous alloys. The strong temperature dependence of the Doppler-broadened annihilation lineshape below room temperature is similar to that found in crystalline metals when positrons are trapped at extended defects like vacancy clusters, voids and dislocation loops [46].

Globaly the results suggest that, in the as-received amorphous state, positrons explore some quenched "empty spaces", less than one atomic volume in size. Similar ideas have been previously presented by Kajcos et al [33] and Cartier et al [47].

These "empty spaces" seem bigger than the empty Bernal holes : the observed positron lifetimes given in table I column 1 are larger than the predicted values for the biggest Bernal holes [25].

A rough estimation, detailed later (§ 3), gives an "empty space" concentration of about 0,1 % in the as received state of amorphous alloys. These "empty spaces" migrate at 100 - 350°C and induce the large broad magnetic relaxation zone observed in this temperature range. A short range ordering occurs, especially in $Fe_{40}Ni_{40}P_{14}B_6$ which enhances the magnetic permeability. Although some of these empty spaces are lost during this migration, there always remains a sufficient concentration for observing a saturated state of the positrons (in the case of iron containing single vacancies, a saturated state is observed for any concentration higher than 5.10^{-5} [48]).

The mobility of these "empty spaces" is distributed around an average value (this is shown by magnetic relaxation : § 2°2.3). However the average size seems to be relatively well defined since it is independent of the cooling rate (§. 2°2.3).

Above the crystallization temperature, the lifetime values remain high because new trapping centers are formed by defects, dislocations, interfaces and grain boundaries. When these trapping centers are eliminated by annealing at higher temperature, the properties of the well annealed alloy are recovered [49].

The experiment on $Pd_{80}Si_{20}$ charged with hydrogen shows that the local electron density of this "empty space" remains unchanged by hydrogenation (§. 2°2.4). This means that hydrogen stays out of the "empty space" center. This suggests that hydrogen is :

- either located at holes too small to be detected by positrons (in iron, the minimal free volume to bind a positron is estimated at 0.5 atomic volume [25]).
- or located as a neighbour of the "empty space".

A similar case exists in fcc metals containing hydrogen and vacancies, where it is shown by channeling experiments, that hydrogen stays in an interstitial site close to the vacancy [50-51].

2°3.2. Irradiation induced defects

The new results show that irradiated amorphous alloys or crystalline metals have distinct features :

- in amorphous alloys, only one positron lifetime is observed, which decreases continuously with the annealing temperature and recovers the initial value after room temperature annealing
- in crystalline metals, two positrons lifetimes can be distinguished in the spectra, provided the irradiation dose does not exceed $\sim 10^{18} e^- cm^{-2}$ (for higher doses, the positron trapping at vacancies is saturated and the spectrum remains the same). The intensity of the longer lifetime recovers by stages well defined in temperature. Sometimes, one of them coincides with a rapid increase of the longer positron lifetime [48, 52, 53].

In irradiated crystalline metals, the following picture of radiation damage is currently accepted [54].

When an energetic particle knocks an atom of the crystalline lattice (with some energetic conditions fulfilled), the atom is ejected from its site and stops elsewhere in the crystal as a self-interstitial, a vacancy is created at the initial site which remains vacant and a Frenkel pair of two anti-defects is formed. It may have several configurations, but two types of pairs are distinguished as a function of the interaction energy between the anti-defects.

- In a "close pair" the interstitial is ejected to a few atomic distances

from the vacancy (less than three atomic distances). The two anti-defects are in strong elastic interaction. There are several distinct configurations due to the distributions of distances and orientations of the pairs [55] (an increase of a few tenth of atomic distances shifts the recovery stages by several degrees [56-57]). The pairs are metastable and thermal fluctuations induce collapse. Discrete recovery steps, referenced as $I_A \ldots I_D$ in the electrical resistivity recovery studies correspond to successive collapse of more and more stable pairs [58]. For instance, in 20 K electron irradiated iron, close pairs form 90 % of the defects created and anneal out between 20 and 100 K [59].

- In other configurations called "uncorrelated pairs", the interstitial is ejected far from its vacancy by a dynamic mass transport process. Different mechanisms are proposed. Some of them are along a straight line and take into account the periodic arrangement (i.e. the "focuson", collective mass transport along a dense row of atoms and the "channeling", individual mass transport in an empty channel). In other mechanisms the propagation is not along straight crystalline rows [60] and such modes could be conceivable in amorphous metals.

In such "uncorrelated pairs", there is no more interaction between the two anti-defects (this interaction could be neglected for interspacing exceeding three atomic distances). When temperature is sufficient, the interstitial can escape without falling into its "mother" vacancy. An isochronal annealing treatment gives rise to several successive recovery stages up to very high temperatures, due to the distinct long range migration and annealing of both constituent parts of the pair [53, 61] :

- stage I_E : self-interstitial long range migration (in iron, the "self-interstitial" migrates at 125 K, making about 1000 jumps during

its half life),

- stage II : interstitial clustering and rearrangement in interstitial clusters (125 - 220 K in Fe),
- stage III : vacancy long range migration and vacancy clustering which induces the increase of the longer lifetime mentioned above (220 K in Fe),
- stages IV and V : disappearance of clusters by several mechanisms (600 - 1000 K in Fe).

Several fundamental and applied investigations into interstitial and vacancy clusters have been undertaken because these clusters induce embrittlement (interstitial clusters) or swelling (vacancy clusters) in nuclear reactor materials.

In amorphous alloys, the new results show clearly that uncorrelated pairs do not exist because neither long range migration nor vacancy clustering are observed. Consequently, dynamic mass transport does not take place in electron or neutron irradiated amorphous alloys. *The hypothesis of close correlated pairs, widely distributed in configurations, explains correctly the results.* This model is now described.

The incident particle ejects an atom in the amorphous alloy. This atom remains at a few distances from the point of ejection. A hole is created. The volume of this hole is approximately the volume of the missing atom (a little smaller because some lattice relaxation occurs). This fact is supported in the four studied alloys, where the positron lifetime at saturation approaches a limit rather close to single vacancy values of the pure metals.

In the hypothesis of a vacancy like defect creation, similar electron irradiation doses are expected to induce, in amorphous Fe_{80}

B_{20} and in crystalline iron, the same order of magnitude of irradiation defect concentration, since the measured experimental threshold displacement energies are very close [43], and the irradiation doses are low enough.

In the amorphous alloys the positron trapping saturation is reached with irradiation doses of one order of magnitude higher than in crystalline metals. This is explained by a positron trapping competition between the pre-existing "empty spaces" and the "irradiation induced holes". As the specific trapping rate of the empty spaces is not negligible, higher concentrations of irradiation defects are needed to saturate positron trapping in the radiation induced holes. Using the trapping model at two defect species [53], a concentration of 0.1 % of pre-existing empty spaces is estimated.

In irradiated amorphous alloys lifetime spectra, it is not possible to discriminate two components corresponding to positrons trapped respectively in pre-existing empty spaces and in irradiation induced holes, because they are too close to each other (for instance in $Fe_{80}B_{20}$: 142 and 170 ps).

The continuous recovery observed with positron lifetime and magnetic relaxation techniques gives additional informations. In the neighbourhood of the "hole", an anti-defect is formed which is a collective interstitial (n+1 atoms in a region where previously n atoms were present). A strong interpenetration between atoms induces high local elastic stresses. The distance between anti-defects is always smaller than the critical distance generating "uncorrelated pairs". The strong interaction between anti-defects makes the pair metastable : the pair is frozen in, but, when a high enough thermal fluctuation

occurs, it collapses after a few atomic oscillations. This explains the difference in stability between pre-existing empty spaces and irradiation induced holes : the first can migrate freely without finding any anti-defects.

The absence of clearly separated recovery stages can be understood by a triple distribution of close pair configurations which smoothes the curves :

- a distribution in the distances between anti-defects, these distances remaining below the critical interaction distance,
- a distribution in the nature of surrounding atoms,
- a distribution in hole sizes (depending on the lattice contraction, the combination with pre-existing empty spaces, the size of the ejected atom...).

After annealing at room temperature, practically all the radiation induced vacancy type defects are removed. This explains why only a slight change in positron parameters have been detected in previous studies of electron irradiated amorphous metals [21-22].

Conclusion

In the as received quenched state of amorphous alloys, the amorphous structure appears to contain "empty spaces" bigger than the Bernal holes. Their size is relatively well defined and smaller than one atomic volume. In the case of $Fe_{80}B_{20}$, the concentration of these "empty spaces" is roughly estimated at about 0.1 %. These "empty spaces" migrate at a temperature below the crystallization temperature, and some are lost ; however their concentration remains high enough to saturate positron trapping. Their migration releases local stresses and could induce short range order like vacancies in crystalline metals but at lower temperatures.

Defects are created in amorphous alloys by low temperature irradiation. They appear like some "close Frenkel pairs" : the ejected atom stays not far from its initial position and is associated to an empty zone, the volume of which being approximatively the volume of the missing atom. The strong elastic interaction between interstitial like and vacancy like defects makes the pair metastable, and recovery occurs by thermally activated collapse. The continuous recovery attests that there is a wide range of pair configurations.

Neither long range migration,nor vacancy clustering are observed.

The recovery of radiation induced damage is completed after annealing at room temperature.

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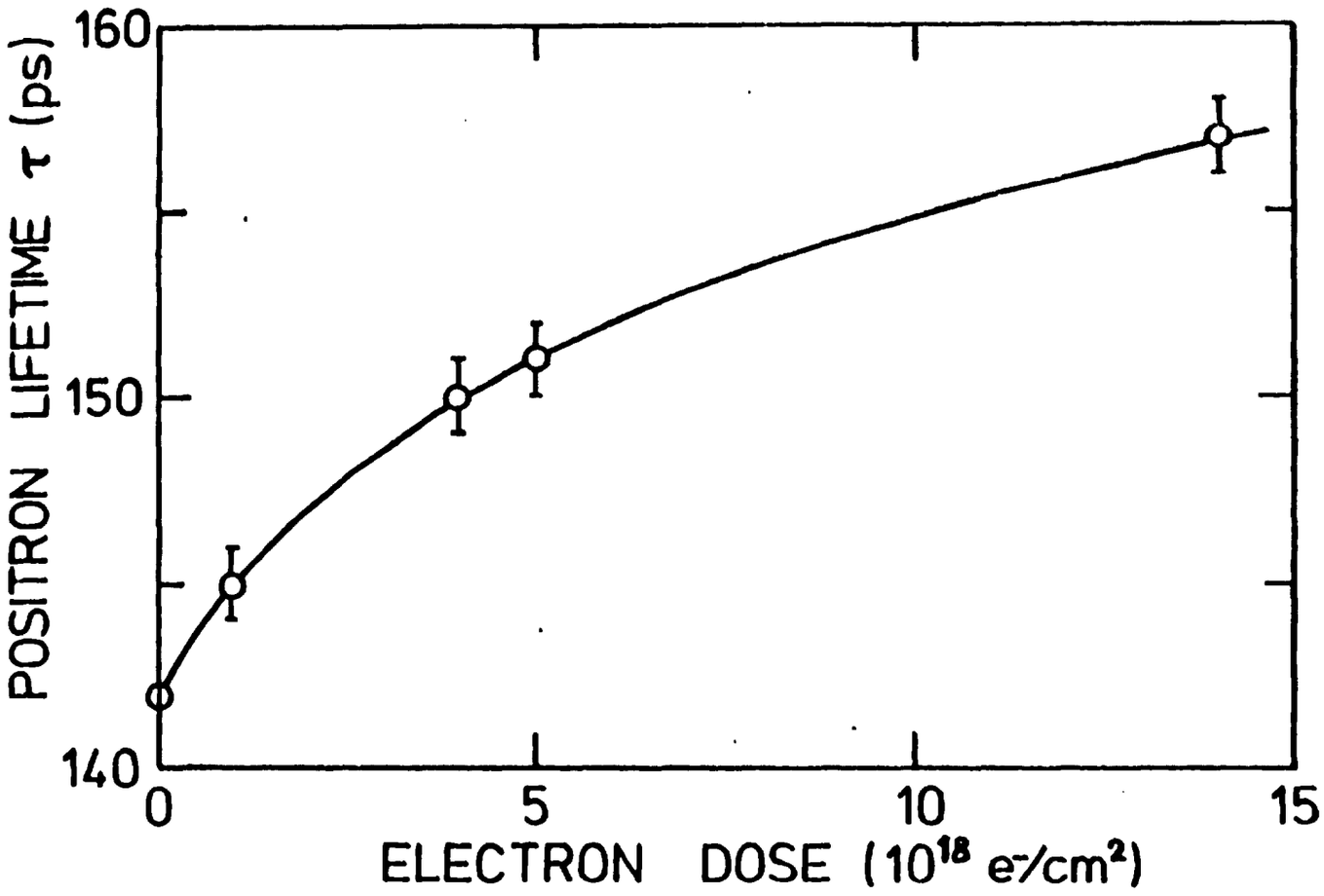
Table I : Positron lifetimes in as received and electron irradiated $Fe_{80}B_{20}$, $Pd_{80}Si_{20}$, $Cu_{50}Ti_{50}$ and $Fe_{40}Ni_{40}P_{14}B_6$ alloys. For comparison characteristic lifetime values in metallic components of the alloys are also shown [2].

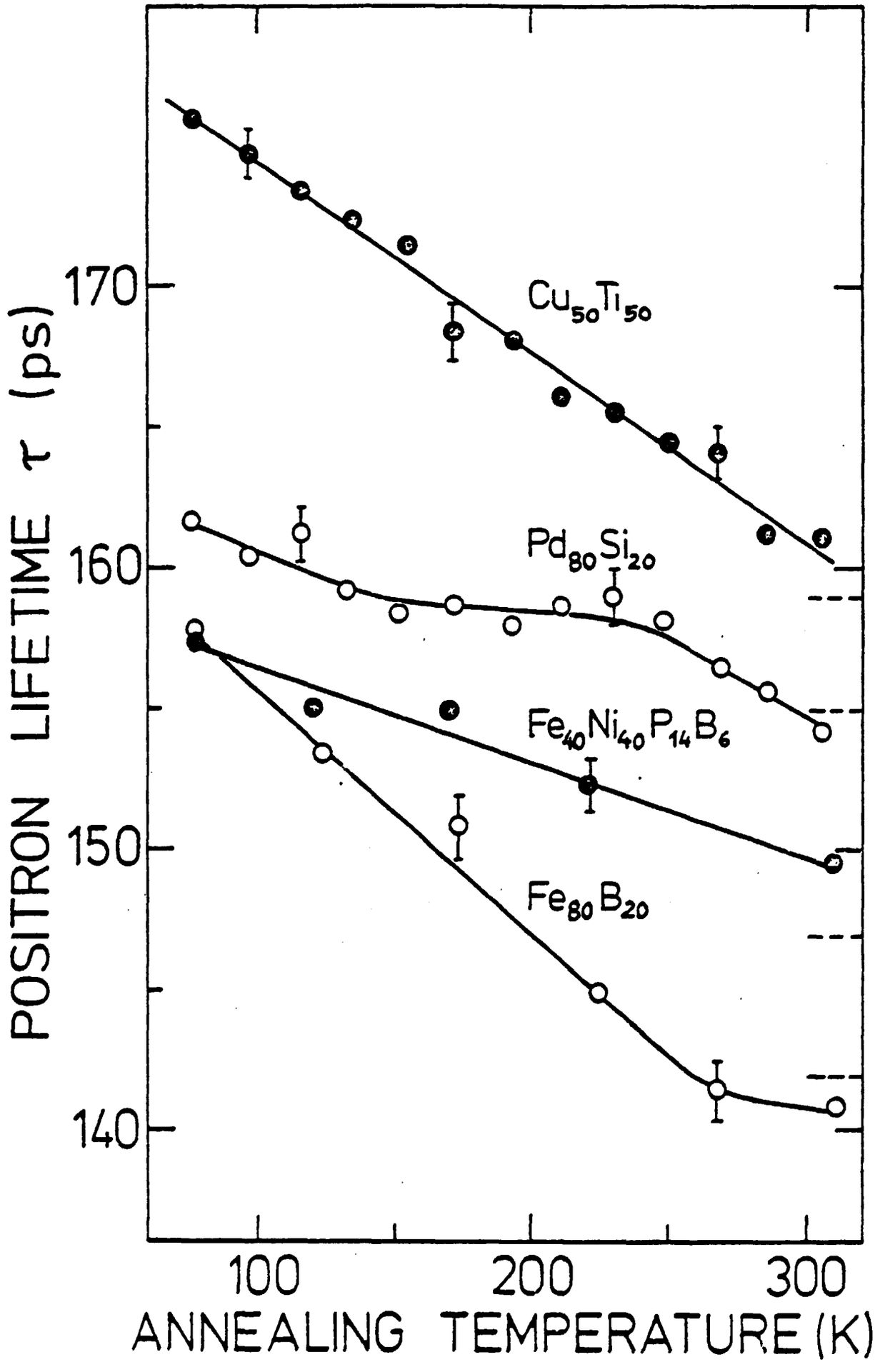
	(1) τ(psec)	(3) τ(psec)
Amorphous	as-received	as-irradiated
$Fe_{80}B_{20}$	142 ± 1	157 ± 1
$Pd_{80}Si_{20}$	155 ± 1	162 ± 1
$Cu_{50}Ti_{50}$	159 ± 1	176 ± 1
$Fe_{40}Ni_{40}P_{14}B_6$	147 ± 2	157 ± 2
Crystal	(2) bulk	(4) vacancy
Fe (present work)	110	171
Ni (ref. 62)	110	180
Pd (ref. 63)	118	-
Cu (ref. 64)	122	180
Ti (present work)	152	222

Figure Captions

Fig. 1 - Positron lifetime as a function of the irradiation dose. Irradiations are performed at 20 K under liquid hydrogen, with 3 MeV electrons. Measurements are made at 77 K [1].

Fig. 2 - Positron lifetime as a function of isochronal annealing temperature in various amorphous alloys electron irradiated at 20 K. Dotted lines in the right margin of the figure represent positron lifetime values in the as-received state of the corresponding alloys [2].


160
150
140
POSITRON LIFETIME τ (ps)
0
5
10
15
ELECTRON DOSE (10^18 e⁻/cm²)


POSITRON LIFETIME τ (ps)
170
160
150
140
Cu50Ti50
Pd80Si20
Fe40Ni40P14B6
Fe80B20
100
200
300
ANNEALING TEMPERATURE (K)