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A mobile positron-lifetime spectrometer for field applications based on β^+-γ coincidence

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Abstract. A mobile positron-lifetime spectrometer based on a β^+-γ coincidence measurement, which has been applied to in-situ examinations of microstructural evolution processes during the fatigue of copper single crystals, is described. Since no sandwich-type geometry is required, it is applicable to all specimen geometries commonly used in materials testing and to the non-destructive testing of engineering parts in service. As a radioactive source ^{72}Se generates the positron-emitting ^{72}As, which provides two positron spectra with maximum energies of 2.5 MeV and 3.3 MeV and a prompt γ quantum of 835 keV. The positrons emitted in the direction towards the specimen pass through a fast plastic scintillator and produce a scintillation signal, thereby losing about 150 keV of their energy. This signal serves as a start signal for the positron-lifetime measurement and is measured in coincidence with the subsequent 511 keV annihilation quantum. After passage through the plastic scintillator the remaining positron energy is still high enough to penetrate deep into the material and to allow for real bulk examinations. The prompt γ quantum may serve as an on-line control of the stability of the electronic system which will be useful under non-constant service conditions in a proposed field application.

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For future application of the highly sensitive method of positron annihilation in non-destructive testing and materials science, an easy, portable instrument is required which serves for different specimen geometries and provides for short measuring times. This should be accomplished with a positron source of moderate activity which allows for safe and simple handling, from the radiological point of view. However, the positron energies should be large enough to access bulk properties with negligible disturbances from surface effects. Accordingly, it seemed highly promising to develop a method of positron annihilation with regard to the requirements of a non-destructive testing technique for engineering parts. A first interesting application in this field may be the in-situ investigation of microstructural evolution processes due to plastic deformation of materials.

With the exception of positron beam techniques [1–3] in current positron-lifetime spectroscopy, γ-γ coincidence measurements are primarily performed using a prompt γ quantum as a start signal and a 511 keV annihilation quantum as a stop signal. With respect to the positron activity of a typical positron source, the γ-γ coincidence rates are low due to the limited detection probability of γ quanta. Thus, a large part of the positrons annihilate without being detected and long measuring times are required. Therefore, the possibilities of in-situ investigations of microstructural evolution processes are limited to processes taking place on a time scale much longer than the measuring time.

If the sandwich geometry is used, two identical specimens or two pieces cut from the same specimen are required, and the positron source is placed between (e.g. [4–6]). From this point of view, the common sandwich technique of positron-annihilation technique is a destructive method.

Typically, ^{22}Na is used as positron source which emits positrons with a maximum energy of 544 keV. These energies yield a typical penetration depth of about 100–200 μm below the material surface. For many applications this penetration depth is too small to investigate the microstructural evolution in the bulk, e.g., since the dislocation density near the surface may be appreciably lower than that in the bulk of a tensile specimen [7–9]. Additionally, in many cases the surface properties of the specimen change during plastic deformation due to the formation of slip lines and slip bands [10, 11]. Therefore disturbing effects on the positron-annihilation properties cannot be excluded.

Three systems for non-destructive testing and damage assessment based on positron-annihilation measurements have been presented in recent literature [14–16].

Somieski et al. [14] measure the positron lifetime by γ-γ coincidence with a sandwich-type geometry. The sandwich consists of the specimen to be analysed, the ^{22}Na source (15 μCi) and a small piece of reference material (GaAs doped with Zn) with a known positron lifetime (230 ps) which also protects the scintillators from direct positron irradiation. The lifetime spectrum has to be corrected for the annihilation events in the reference material.

Hutchings et al. [15] use a ^{68}Ge source of 1 mm^2 size (20 μCi) housed in an aluminium holder. It is reported that the holder is specially designed to maintain a good spatial resolution and that half of the positrons annihilate there, giving a constant contribution to the line shape. Since the line-shape parameter S is measured with a germanium detector, no coincidence is required, the counting rates are high and the geometry is rather simple. The ^{68}Ge emits positrons with an energy of up to 1.9 MeV which makes real bulk properties accessible due to the larger penetration depth into the material as compared to ^{22}Na. Uchida et al. [16] also developed a portable annihilation line shape analysis system with a ^{68}Ge source which has been applied to assess the fatigue damage in stainless and low alloy steels and the microstructural changes during aging of duplex stainless steels.

The present paper introduces a method of non-destructive testing which appreciably reduces the background and allows for coincidence rates of life-time measurements in the kHz range using positron activities of about 30 μCi. The method is based on an idea of Graham and Bell [12] and of Maier and Myllylä [13], that is to use a β^+-γ coincidence instead of the γ-γ coincidence. This is carried out by placing a thin, fast plastic scintillator between the source and the specimen. Thus, positrons being emitted from the source pass the scintillator and lose about 150 keV of their initial energy which provides the start signal.

1 Set-up of the β^+-γ spectrometer

Figure 1 shows that ^{72}Se as the parent nucleus decays by electron capture into ^{72}As which undergoes a β^+ decay emitting two β^+ spectra with maximum energies of 2.5 MeV and 3.3 MeV. Since the half-life of ^{72}Se is 8.4 days and that of ^{72}As is only 26 h, a radioactive equilibrium is established after 3.6 days which leads to a β^+ activity decreasing with a half-life of 8.4 days. The short half-life minimizes the risks of eventual contamination which is important for field applications of the portable set-up. The emission of a positron of 2.5 MeV is followed by the emission of a prompt γ-quantum of 835 keV which serves as a stop signal in order to record an on-line prompt spectrum.

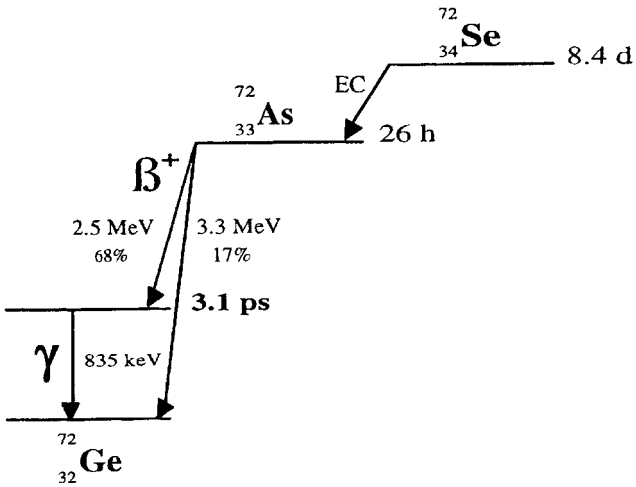

Fig. 1. Decay scheme of ^{72}Se after [17]. Note the prompt γ radiation of 835 keV energy of the ^{72}Ge

The source isotope, ^{72}Se, was produced by irradiating a germanium target with α-particles of 55 MeV energy at the Bonn cyclotrone. The irradiated germanium target was melted in a graphite crucible. Since the vapour pressure of selenium at 1250 K is 10^{13} times higher than that of germanium [18], the selenium isotopes can easily be separated from the target material. The ^{72}Se vapour could escape from the crucible only through a small hole as indicated in Fig. 2. The vaporized selenium condensed preferentially on the inner surface of a cooled (270 K), cylindrical gold collimator which was closely placed above the graphite crucible. The whole set-up was placed in a vacuum vessel at a pressure of 5×10^{-5} mbar [19].

The collimated source is covered by a fast plastic scintillator of one millimeter thickness[1] which has to be passed by each positron which contributes to the lifetime measurement (see Fig. 3). The effect of the gold collimator (inside diameter 0.6 mm, outside diameter of 2.0 mm) depends on the spatial distribution of the selenium inside. Positrons which are emitted by atoms on the bottom of the collimator will either

[1] BC 418 from BICRON Organic Scintillators

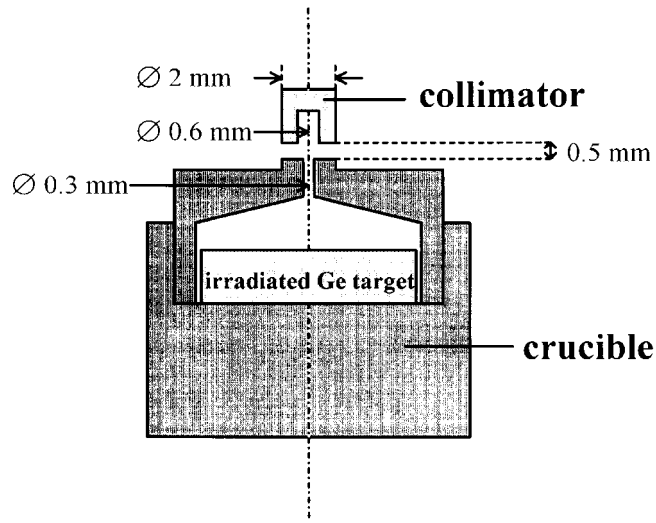

Fig. 2. Preparation of a ^{72}Se source in a gold collimator by vapour deposition. Figure after Tongbhoyai [19]

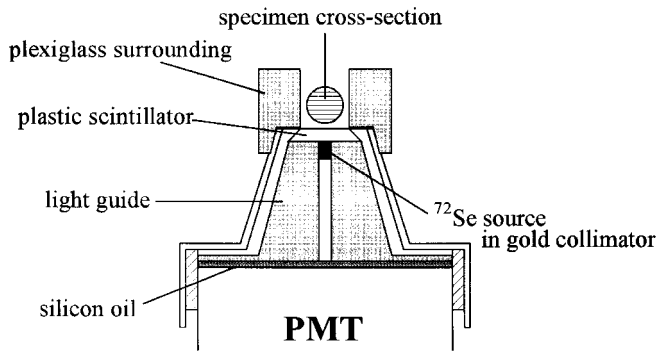

Fig. 3. Start detector set-up consisting of the fast plastic scintillator, the plexiglass light guide the ^{72}Se collimated source, and the photomultiplier tube. The specimen itself is surrounded by Plexiglass

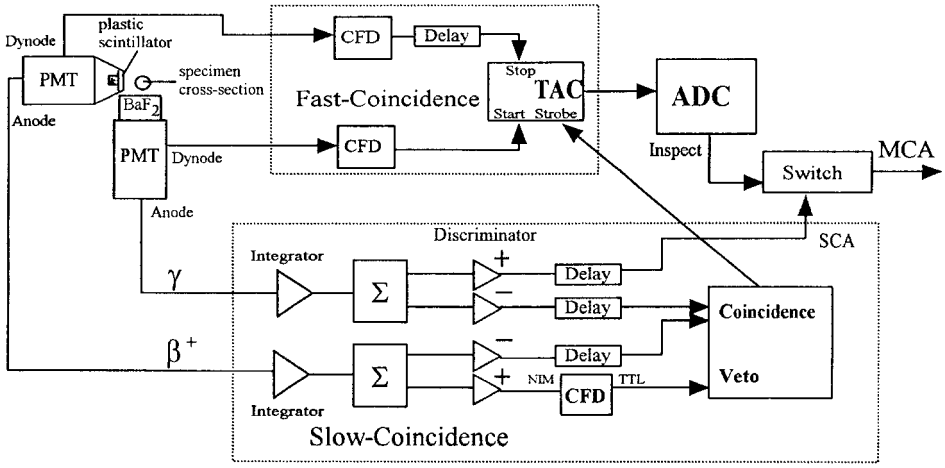

Fig. 4. Scheme of the electronic circuits. (CFD constant fraction discriminator; TAC time-to-amplitude converter; ADC analog-digital converter; MCA multi channel analyzer; PMT photomultiplier tube). The input signals for the fast coincidence circuit are supplied by the 8th dynode of the 12-stage photomultiplier tubes (Philips XP 2020)

directly hit the specimen or annihilate in the gold. Positrons which are emitted from atoms on the upper part of the interior surface may miss the specimen and annihilate elsewhere. However, in this case, the energy loss of the positrons in the scintillator is higher than the 150 keV for the direct, perpendicular passage. Therefore, in order to reduce the background, the collimating effect may be supported electronically by requiring a maximum energy loss close to 150 keV.

The collimator-source set-up is housed in the tip of a plexiglass cone which serves as a light guide and is placed on a fast photomultiplier tube (Philips XP 2020). In order to allow an easy exchange of exhausted positron sources, the optical contact between the Plexiglas cone and the photomultiplier tube (PMT) is of silicon oil. The whole arrangement is placed in a light-proof aluminium container with a thin aluminium foil of 20 μm thickness acting as the positron window.

Since the half-life of only 8.4 days limits the duty cycle, an initial positron activity of 30 μCi is chosen, which gives an initial β^+-γ coincidence rate of several thousands per second.

Figure 4 shows a schematic layout of the data-aquisition system which is a standard lifetime measurement system with slight modifications. Since the detection efficiency of a γ quantum in a BaF_2 detector is much lower than the 100% probability to detect the passage of a positron in the plastic scintillator, in practice, inverted lifetime spectra are recorded. The signal of the BaF_2 detector is used as the start signal and the delayed signal of the plastic scintillator serves as a stop signal [13]. In this way the time-to-amplitude converter is not charged with positron-passage signals which will not find their corresponding annihilation signal. Hence, the dead time is reduced and the set-up is able to cope with coincidence rates of many kHz.

The 511 keV annihilation quanta and the 835 keV prompt quanta are detected with the aid of a $1.5''BaF_2$ detector. A discriminator unit is adjusted to distinguish between 511 keV and 835 keV quanta and to trigger a switch for the data aquisition in a multi channel analyser (cf. Fig. 4). So, the positron-annihilation and the prompt line are recorded simultaneously. The latter allows an on-line control of the time

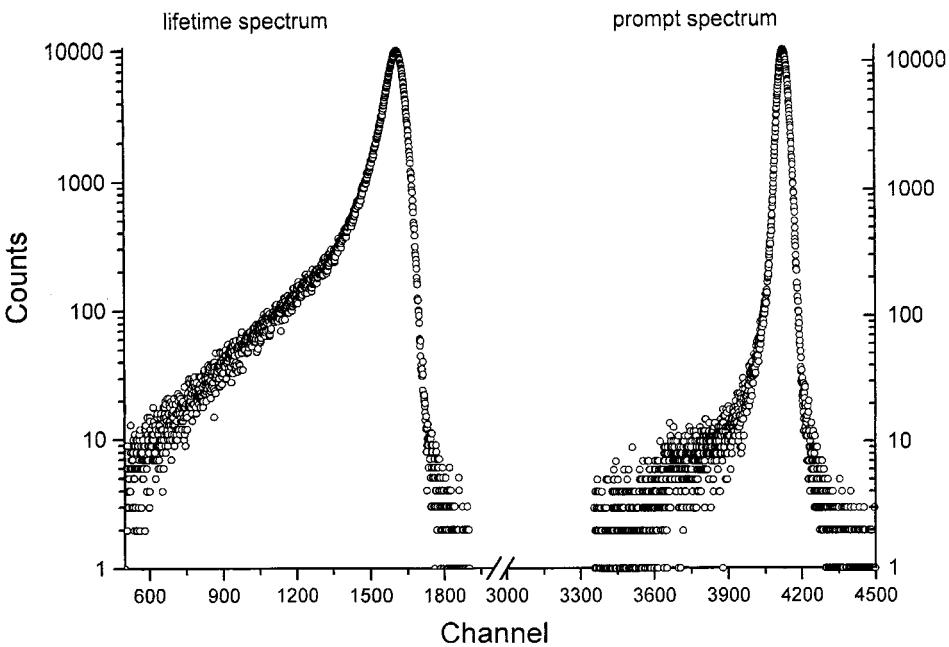

Fig. 5. Example for a simultaneously recorded (inverse) positron-lifetime spectrum and the prompt 835 keV γ spectrum. The full width at half maximum of the prompt curve allows for an on-line control of the time resolution of the spectrometer and yields a resolution of 280 ps

resolution of the spectrometer which is measured using the start signal of a 2.5 MeV positron and the successive 835 keV γ quantum as the stop signal. This is especially helpful in monitoring the stability of the electronic components under variable environmental conditions. The full width at half maximum of the prompt line yields values of 280 ps. Figure 5 gives an example of a simultaneously recorded inverse positron-lifetime spectrum and the prompt curve.

2 In-situ positron-lifetime measurements during fatigue experiments

The positron-lifetime spectrometer was used for an on-line assessment of fatigue damage in cyclic deformation experiments with copper single crystals. The following example is part of an extended investigation of the applicability of positron annihilation to the non-destructive characterization of defect structures in plastically deformed metals and alloys. In order to meet our requirements, a small closed-loop controlled cyclic deformation machine was constructed which is driven by a piezoelectric translator. Currently, total strain amplitudes up to 50 μm and force amplitudes up to 2 kN can be achieved [20, 21].

The cyclic deformation experiments were performed in symmetric push-pull mode under total strain control with a frequency of 2 Hz. Mechanical hysteresis loops were recorded and the cyclic hardening curve was determined. The plastic shear strain amplitude resolved to the primary glide plane, $\hat{\gamma}_{\mathrm{pl}}$, was determined from the plastic strain in the hysteresis loops at zero force. The cumulative plastic shear strain $\gamma_{\mathrm{cum}} = \sum_{\mathrm{i}} 4N_{\mathrm{i}}\hat{\gamma}_{\mathrm{pl,i}}$ was calculated where N_{i} denotes the number of deformation cycles with $\hat{\gamma}_{\mathrm{pl,i}}$ The resolved shear-stress amplitude $\hat{\tau}$ was determined from the mean value of the amounts of the maximum and minimum force in tension and compression. Figure 6 shows the resulting cyclic hardening curve (For details of typical fatigue experiments see e.g., [22]) and the results of the in-situ measurement of the mean positron lifetime $\overline{\tau}_{\mathrm{e^+}}$.

In our experiments cylindrical single crystals have been used, approximately 50 mm long and 5 mm in diameter, with a central gauge length of 10 mm length and 4 mm in diameter. Even if the collimated source set-up is placed at a distance of 1 mm from the specimen, about 50% of the positrons which produce a valid start signal in the scintillator miss the specimen and annihilate elsewhere. As far as possible the specimen was surrounded by plexiglass in order to allow these positrons to annihilate only in a material in which positronium forms. Since the pick-off annihilation of the positronium triplet state contributes to the lifetime spectra with a significantly longer lifetime (≥ 1.5 ns) it can be distinguished from annihilation events in the copper specimen. Thus, the background in the recorded spectra is mainly caused by positrons annihilating either in the plexiglass surrounding the specimen or in the plastic scintillator. Before each experiment a pure background spectrum is accumulated by placing a plexiglass specimen in the spectrometer. No additional lifetime components are introduced, since the plexiglass and the plastic scintillator exhibit the same positron lifetime spectra. However the percentage of positrons which are backscattered by the specimen may differ from the conditions during the experiment when metallic specimens are examined. But only the small portion of positrons that additionally annihilate in the thin aluminium foil acting as positron window yield a difference between the measured and the real background.

Before the mean positron lifetime is determined, the background can be removed from the spectra by a weighted subtraction of the recorded pure background spectrum. Therefore we define a fixed window that clearly lies within the range of the long-lived component of the spectra. In this way the weight coefficient is given by the ratio of counts accumulated inside this window in the examined spectrum and in the pure background spectrum. After the subtraction of the background, a numerical fit by a weighted linear regression yields

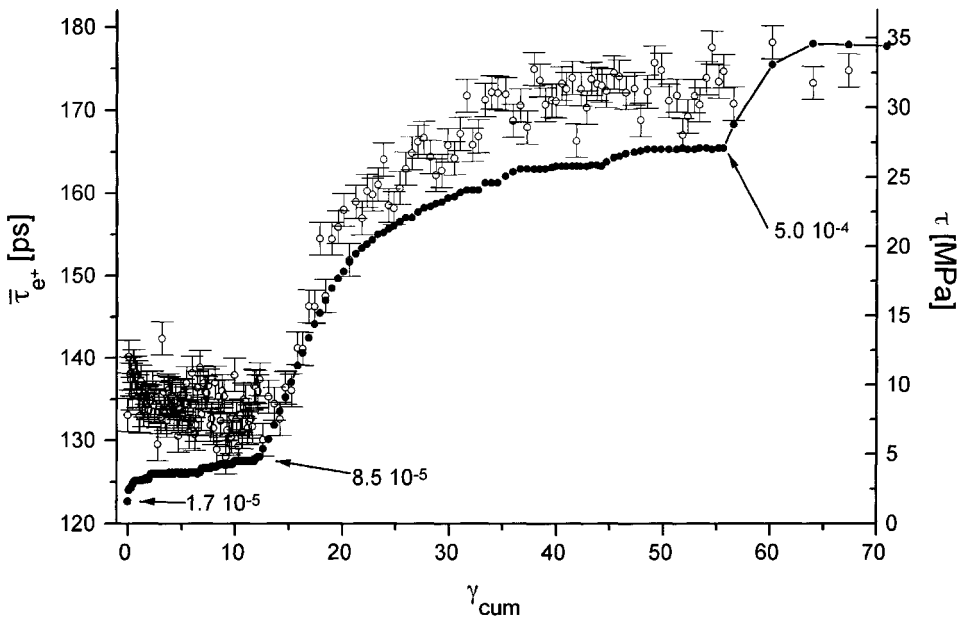

Fig. 6. Cyclic hardening curve, i.e., resolved shear-stress amplitude $\hat{\tau}$ versus the cumulative resolved shear strain γ_{cum} (symbol •) of the copper single crystal Cu-79 and the mean positron lifetime $\overline{\tau}_{\mathrm{e^+}}$ (symbol ○). The arrows indicate an increase of the applied total strain amplitude to the values indicated

the mean positron lifetime[2]. The precision of $\overline{\tau}_{e^+}$ depends on both the stability of the electronic devices and the accuracy of the applied evaluation procedure. A repeated measurement performed on an undeformed specimen showed a statistical scatter of the obtained mean lifetimes of less than ± 2 ps. This is, however, three times more than the statistical error due to linear regression fit of the lifetime spectrum after correction for the background. The absolute values of the mean positron lifetime obtained from several undeformed crystals can be reproduced within ± 5 ps.

At small amplitudes of the resolved plastic shear strain ($1.7 \times 10^{-5} \leq \hat{\gamma}_{pl} \leq 8.5 \times 10^{-5}$), up to cumulative strains of 15, no significant extension in the positron lifetime is observable. After an increase of $\hat{\gamma}_{pl}$ to 8.5×10^{-5} a pronounced cyclic hardening sets in, which is accompanied by an increase in the positron lifetime. The increase of the positron lifetime takes place just in a range of $\hat{\gamma}_{pl}$ where the mesoscopic dislocation arrangement in face-centred cubic metals undergoes severe changes. In detail, the so-called *matrix* structure which is characterized by a constant, static dislocation density becomes instable and is partially replaced by so-called *persistent slip bands* (PSBs).[3]The constant dislocation density in PSBs, however, results from a dynamic equilibrium between production and annihilation of dislocations which produces high concentrations of atomic defects. A further increase of $\hat{\gamma}_{pl}$ to 5.0×10^{-4} shows no further increase of $\overline{\tau}_{e^+}$ which indicates a saturation effect. A detailed interpretation of our results will be given elsewhere.

For our purpose, i.e., to test the utility of in-situ positron-lifetime measurements, it is important to note that the microstructural changes are reflected both in the cyclic hardening curve ($\bullet$ in Fig. 6) and in the corresponding development of the mean positron lifetime ($\circ$ in Fig. 6). The cyclic deformation procedure did not need to be stopped for recording the positron-annihilation spectra. The reduction of the sampling time to several minutes already allows for an in-situ detection of microstructural changes at typical frequencies in strain controlled fatigue experiments (≤ 2 Hz).

3 Discussion

The lifetime spectrometer described above is improved with respect to conventional γ-γ coincidence systems, by using the much higher detection efficiency of positrons in a plastic scintillator. Therefore, the β^+-γ coincidence rates are approximately ten times higher than the γ-γ coincidence rates in a conventional set-up with the same source. This is a prerequisite of in-situ assessment of deformation damage. Since the positrons which annihilate in the collimated source do not make any contribution to the spectrum, because they do not produce a start signal, the background is reduced appreciably. Only the positrons which annihilate in the plastic scintillator and those which are missing the specimen and annihilate elsewhere contribute to the background. However, if the positrons missing the crystal are only allowed to annihilate in the plexiglass which surrounds the specimen, the background can be reduced by subtracting a plexiglass reference spectrum. If we use a copper plate instead of a cylindrical specimen, the geometry becomes similar to the one used in [14] and [15]. In this case the background is reduced to about 30% which is much lower than the 44% and about 50% cited in [14] and [15], respectively. The inversion of the start and stop signals helps to avoid effects of a counting-rate saturation and allows us to cope with the high β^+-γ coincidence rates [13].

Usually, standard methods in non-destructive materials testing (e.g., ultrasonics) require portable equipment in order to test engineering parts that are actually in service. This type of investigation takes place under different environmental conditions and the results have to be reproducible and comparable. This means that the absolute values of $\overline{\tau}_{e^+}$ have to be reproducible for materials in the same stage of fatigue. Therefore, one has to aspire to work with a well defined detector geometry, a constant adjustment and a constant temperature of the electronic set-up presented in Fig. 4. The proposed system provides the possibility of an on-line control of the stability of the electronic set-up and the detectors by recording the 835 keV line of prompt γ quanta. Hence, the proposed system satisfies all major requirements for a portable positron-annihilation equipment in non-destructive testing. The necessity to install a new positron source every few weeks due to the short half-life may be balanced by safety requirements when radioactive substances are handled outside laboratories.

For metals and alloys with a density of $9\,g/cm^3$ the positron energies, after passage through the start scintillator of up to 2.3 MeV and 3.1 MeV are sufficient for the majority of the positrons to penetrate into the specimen to a depth of more than 250 μm and 400 μm, respectively.[4] This penetration depth enables one to access real bulk properties and to study the evolution of defect structures during plastic deformation.

Only a few similar studies of fatigue damage accumulation using positron annihilation have been reported. Due to the intended technological applications the S parameter was measured for an investigation of steels [15, 16] and superalloys [25]. In order to reveal basic features of fatigue and fatigue crack initiation, positron-lifetime studies are reported on copper and nickel crystals [26, 27]. The positron studies on copper have been performed on a series of single crystals and polycrystals which were cyclically deformed under identical conditions until different stages of fatigue were reached [26]. The specimens were then sliced in order to obtain identical parts for the sandwich geometry. Different stages of fatigue damage accumulation were discernible in sandwich specimens cut from different crystals. Since this method is destructive, one has to know when to stop the cyclic deformation experiments, i.e., some knowledge about the dynamics of the fatigue damage accumulation is required. Otherwise, a large number of fatigued specimens is necessary and the investigation may become rather cumbersome.

[2]By the use of a ^{54}Mn source, which provides γ radiation of 835 keV energy, it has been measured that about 15% of the prompt 835 keV quanta are Compton-scattered in the BaF_2 detector and will erroneously be counted as 511 keV quanta. These events, however, measure the lifetime of the excited state of the ^{72}Ge of 3.1 Ps (cf. Fig. 1). Therefore, in the determination of $\overline{\tau}_{e^+}$ this will cause a somewhat smaller absolute value. Nevertheless, since the conditions of recording and evaluating the spectra are constant, this shortage of $\overline{\tau}_{e^+}$ will be approximately the same for all values and the effects on the interpretation of our results are negligible.

[3]For further information on the underlying features of cyclic deformation the reader is referred to some recent publications, e.g., [23, 24].

[4]The most probable positron energy ($\approx 1/3 E_{max}$) in the spectra is used to calculate the penetration depth. The maximum available energy of a β spectrum has negligible probability and the annihilation of the corresponding positrons does not significantly contribute to the signal.

Real in-situ experiments have been performed on nickel crystals [27]. The nickel was irradiated with protons in order to produce ^{58}Co as an interior positron source. However, this elegant technique is limited to nickel and nickel-rich alloys and not suitable for our purposes, i.e., to search for a standard method based on positron annihilation.

A final remark concerns the evaluation of the obtained positron-lifetime spectra. Often, the spectra are analysed by fitting several lifetimes which contribute to the mean lifetime with a certain statistical weight or "intensity" [26, 27]. This procedure is a valuable tool if it is certain that only a small number of well known types of positron traps are present. If we, however, consider the spectrum of different types of defects in fatigued metallic specimens (dislocations, jogs on dislocations, vacancies, di-vacancies, vacancy clusters of variable size, dislocation debris), a decomposition of the mean lifetime seems quite risky. For example, so far no physical reason can adequately explain why the vacancy clusters (microvoids) formed during fatigue should have a discrete and well defined size. Rather they obey a certain but unknown size distribution. In this case, according to calculations of Hautoärvi et al. [28] the positron lifetimes are a function of the radius of the microvoids. Whereas monovacancies and divacancies will be easily discernible, microvoids containing 5 to 50 vacancies will contribute with increasing lifetimes to the spectra which presumably cannot be resolved. Nevertheless, in the case of fatigue two lifetimes were often fitted to the spectra, denoting dislocations and microvoids. Since the physical significance and the practical enforcement of such a procedure appears to be questionable at the present state of knowledge, we prefer to trust the mean positron lifetime.

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