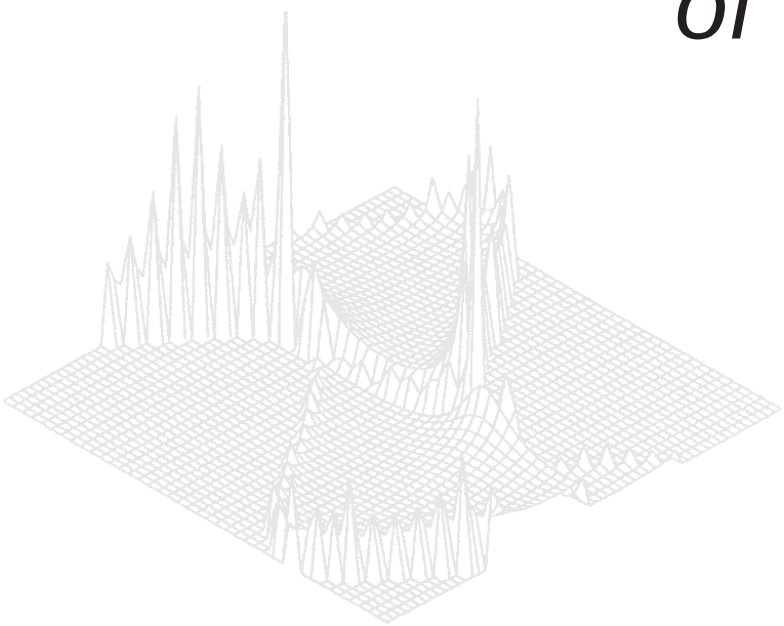
CSIRO PUBLISHING

Australian Journal of Physics

Volume 53, 2000



A journal for the publication of
original research in all branches of physics

www.publish.csiro.au/journals/ajp

All enquiries and manuscripts should be directed to
Australian Journal of Physics
CSIRO PUBLISHING
PO Box 1139 (150 Oxford St)
Collingwood Telephone: 61 3 9662 7626
Vic. 3066 Facsimile: 61 3 9662 7611
Australia Email: peter.robertson@publish.csiro.au

Published by **CSIRO** PUBLISHING
for CSIRO and
the Australian Academy of Science

Aust. J. Phys., 2000, **53**, 829–834

Positron Lifetimes and Microhardness in Thermal Fatigued 4Cr5MoSiV Steel

C. Q. Tang,[A] *Z. C. Xia,*[A] *X. Z. Li*[B] *and B. T. Yao*[B]

[A] Department of Physics, Huazhong University of Science and Technology, Wuhan 430 074, People's Republic of China.
[B] Department of Materials Science, Huazhong University of Science and Technology, Wuhan 430 074, People's Republic of China.

Abstract

Positron lifetimes and microhardness have been measured as a function of the thermal fatigue cycle number N in 4Cr5MoSiV steel. It is found that with increasing N: (a) the positron lifetime parameters τ_1, τ_2, I_1, I_2 and τ and the microhardness parameter MH all exhibit quasi-periodic up-and-down variation; (b) these parameters have the same period of variation; and (c) the period of up-and-down variation becomes gradually longer. The variation of MH reveals that fatigue hardening and fatigue softening occur alternately in the process of thermal fatigue. The variations of positron lifetime parameters reveal variations of defects in the fatigued materials. These variations are attributed to microdeformation and dynamic recovery dominating alternately in the process of thermal fatigue.

1. Introduction

It is well known that positron annihilation lifetime spectroscopy (PALS) is a useful tool for studying lattice defects in metals and alloys (Hautojarvi 1979; Brandt and Dupasquier 1983). PALS has been widely used to study variations of structure and defects induced due to quenching, deformation, fatigue, radiation etc. in many metals and alloys. Due to the complexity of data interpretation most of the earlier positron annihilation studies on defects were limited to simple metals and alloys rather than complex polycrystalline materials. However, probing defects and their variations in a complex material and/or in a material undergoing a complex treatment process is a more attractive subject and is also challenging to PALS.

Thermal fatigue is an important phenomenon for a heat-worked die material. In the process of thermal fatigue, the die material undergoes periodic heating and cooling, and so is damaged to different degrees. Usually, the fatigue damage is evaluated by observing crack networks at the surface of the fatigued material; however, these crack networks reflect only surface damage and cannot reveal interior damage information of the fatigued sample. Our work on positron lifetimes in thermal fatigued die steels (Tang *et al.* 1993*a*, 1993*b*, 1993*c*) has shown that PALS may be a useful tool for probing variation of defects in the interior of fatigued material. However, numerous subjects such as the determination of defect type in complex alloys, corresponding relationships between analysed positron lifetimes and real positron lifetimes in the case of high defect-concentration, and the utility of PALS in relation to other techniques, should be investigated further.

In the present work we perform a detailed study of positron lifetime spectra and microhardness in a die steel, 4Cr5MoSiV, which has undergone thermal fatigue. In this paper we report and discuss these new experimental results.

 10.1071/PH00041 0004-9506/00/060829$05.00

2. Experiments

The material used in the experiment was 4Cr5MoSiV die steel. In addition to the Fe and C, the composition of this steel is Cr (4.88 wt%), Mo (1.20 wt%), Si (1.02 wt%) and V (0.93 wt%). The steel was cut into bars with dimensions of $25 \times 10 \times 10\,\mathrm{mm}^3$. These bars underwent heat treatment as follows: quench into oil at room temperature from 1050°C, then temper at 600°C for 2 h and at 500°C for 1.5 h. After heat treatment, all bars were thermally fatigued to a different degree, using the self-constrain Uddeholm method (Maim and Norstrom 1979). In this process, the steel bar is alternatively heated by high-frequency induction and cooled by using compressed air carrying water fog between two different temperatures (here 200°C and 700°C). Each continuous heating and cooling is termed one cycle. The fatigue cycle number is referred to as the fatigue number N. In each cycle the heating and cooling time is a few seconds respectively (here 4 s for heating and 6 s for cooling). It was found that with increasing N, cracks formed and became gradually networked at the surface. In order to investigate the variation of defects in the interior of the material, a couple of samples measuring $25 \times 10 \times 1\,\mathrm{mm}^3$ were cut from opposite surfaces of each fatigued steel bar and were used in the positron lifetime and microhardness measurements.

The positron lifetime spectra were measured at room temperature by using a fast–fast coincidence lifetime spectrometer with a time resolution (FWHM) of 265 ps. In the measurement the positron source, ^{22}NaCl of about 20 μCi enclosed by two pieces of thin mylar foil ($1.2\,\mathrm{mg\,cm}^{-2}$), was sandwiched between each pair of samples mentioned above, and the surface of each sample during fatigue was facing the source. By measuring a positron lifetime spectrum in a pure silicon sample, the source component was determined to have a lifetime of 1.5 ns and an intensity of 4.5%. After source correction the lifetime spectra were resolved with three exponential components and the lifetimes τ_1, τ_2 and τ_3 and their relative intensities I_1, I_2 and I_3 were obtained. Following PALS measurements, the microhardness of the samples was measured.

3. Results and Discussion

In the resolved positron lifetimes, the uncertainty in τ_1 is 3 ps or less, and the uncertainty in τ_2 is 12 ps or less, for all samples. And a value of τ_3 of 1–2 ns with I_3 of ∼4% is regarded as the positron lifetime at the surface of the samples, and thus will not be discussed here.

It is known that when there are many kinds of defects in a sample, the resolved positron lifetime components decomposed from a lifetime spectrum may be weighted averages of some actual lifetimes that are too close to resolve. Thus, it is difficult to clarify a defect state to which each resolved lifetime component corresponds. For such a case, the average positron lifetime may be a suitable parameter to describe the total defect behaviour. In practice, the average positron lifetime is not only the weighted average value of lifetimes in all positron states in a sample, but is also the weighted average value of all resolved lifetimes from the lifetime spectrum. In the present fatigued samples there are doubtless many kinds of defects such as dislocation, vacancy, void (including vacancy clusters) etc. Thus, in order to obtain some information relating to the total defect behaviour in the experimental samples from the positron experiment, the average positron lifetime τ for every lifetime spectrum is found from

$$\tau = (I_1\tau_1 + I_2\tau_2)/(I_1 + I_2). \tag{1}$$

The average positron lifetime τ and the microhardness parameter MH for all samples as functions of the fatigue number N are shown in Fig. 1. The positron lifetimes τ_1, τ_2 and their relative intensities I_1, I_2 for all samples as functions of N are shown in Fig. 2. It is easily seen from Figs 1 and 2 that with increasing N the variations of τ, MH, τ_1, τ_2, I_1 and I_2 demonstrate the following common characteristics: (a) all parameters exhibit quasi-periodic up-and-down variation; (b) these parameters have the same period of variation; and (c) the period of up-and-down variation becomes gradually longer. It is necessary to point out that the variation of τ_1 and τ_2 is independent but I_1 and I_2 always vary inversely as the sum of I_1 and I_2 is always constant. These characteristics are similar to those in the

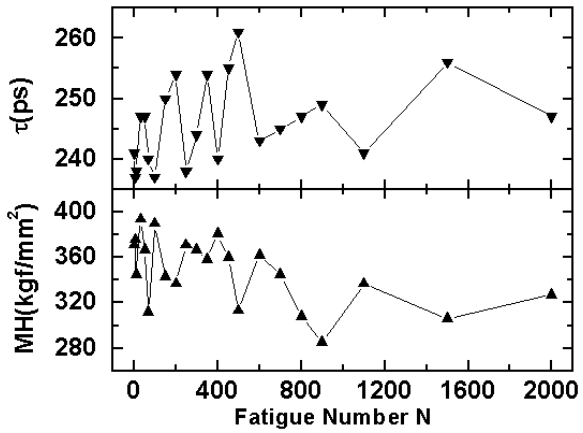


Fig. 1. Average positron lifetime τ and microhardness parameter MH as functions of the fatigue number N for 4Cr5MoSiV steel.

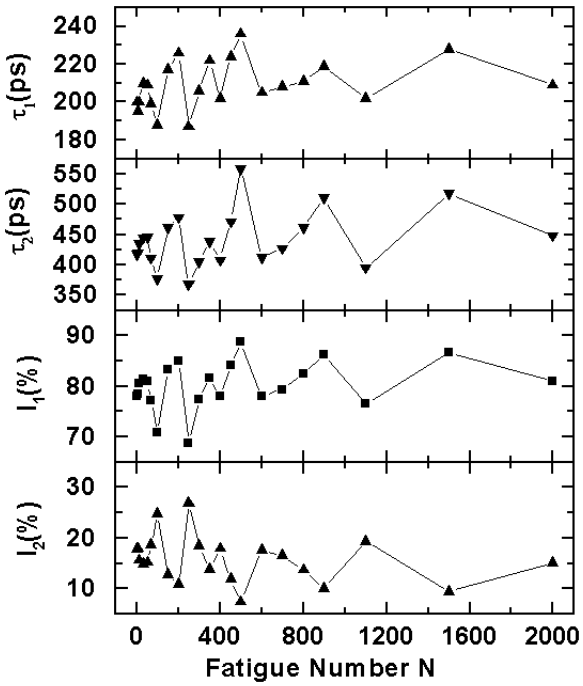


Fig. 2. Positron lifetimes τ_1 and τ_2 and their relative intensities I_1 and I_2 as functions of the fatigue number N for 4Cr5MoSiV steel.

thermal fatigued 5Cr2NiMoVSi and 3Cr2W8V steels (Tang *et al.* 1993*a*, 1993*b*, 1993*c*). For the convenience of discussion, each up-and-down variation is divided into two stages: stage A in which τ_1, τ_2, I_1 and τ are decreased and I_2 and MH are increased and stage B in which τ_1, τ_2, I_1 and τ are increased and I_2 and MH are decreased.

For variation of the positron lifetime parameters with N, it is noteworthy that such quasi-periodic up-and-down variation has not been observed in earlier studies of positron annihilation on mechanical fatigue in which the average positron lifetime and the parameter S in the Doppler broadening measurement of the annihilation γ-rays energy monotonically increases with increasing number of fatigue cycles (see e.g. Karjalainen *et al.* 1982; Grobstein *et al.* 1985). The difference in variation of the positron annihilation parameters between thermal fatigue and mechanical fatigue may result from a different fatigue mechanism and influence on materials of the two fatigue methods. In the earlier studies of positron annihilation on mechanical fatigue, the materials underwent high-temperature annealing and so had low defect concentration prior to fatigue. In addition, materials underwent only tensile fatigue, which caused the defects in the material to increase gradually with N. Thus, the average positron lifetime in these materials became gradually longer. However, in the case of thermal fatigue studied here, the conditions are different. Firstly, before thermal fatigue, the materials contain a very high defect concentration because they underwent a complex treatment, such as forge, quench and temper. Secondly, the thermal fatigue process uses alternative heating and cooling to cause non-uniform expansion and shrinkage of material, and can thus cause microdeformation of materials. Thirdly, in the thermal fatigue process the fatigued material lies always at a higher temperature between 200°C and 700°C, and such a higher temperature is favourable for dynamic recovery. These three conditions are important for thermal fatigue.

The up-and-down variation of MH (see Fig. 1) indicates that fatigue hardening and fatigue softening occur alternately in the process of thermal fatigue. In general, in metals and alloys the density and structure of dislocations are important contributors to the hardness of materials, and high density of dislocations and fixed dislocation structure will give higher hardness to material. Inversely, a low density of dislocations and relaxed dislocation structure can introduce a lower hardness of material. For the present thermal fatigued samples, in stage A, an increase of MH should result from an increase in dislocation density caused by microdeformation in the material. In stage B dynamic recovery results in relaxation of dislocations, this relaxation makes fixed dislocations move anew and cancel each other out, thus decreasing dislocation density and microhardness in the material.

From Fig. 1 it is seen that τ and MH have an opposite variation, i.e. every increase of τ corresponds to a decrease of MH, while every decrease of τ corresponds to an increase of MH. Such a relation between τ and MH may result from different actions of defects on τ and MH in thermal fatigued samples. Because τ is the weighted average value of lifetimes in all positron states in a sample, when the concentration of defects with shorter positron lifetimes increases and/or the concentration of defects with longer positron lifetimes decreases, τ will decrease; conversely, when the concentration of defects with longer positron lifetimes increases and/or the concentration of defects with shorter positron lifetimes decreases, τ will increase. As mentioned above, an increase of MH results from an increase of dislocation density and a decrease of MH is associated with a decrease of density. In general, in the same material the positron lifetime for dislocations is shorter than it is for vacancies and voids (Mackenzie 1983). In the measured thermal fatigue samples there should be a large amount of dislocations, vacancies, voids etc., so for a variation of τ one possible contribution is a variation of dislocation density. In stage A, an increase in

the dislocation density could cause a decrease of τ, and in stage B a decrease of dislocation density results in an increase of τ.

In the above discussion on τ and MH the behaviour of vacancies and voids is not revealed. Their behaviour is related to the variations of the lifetimes τ_1 and τ_2 and their relative intensities I_1 and I_2 (Fig. 2) and will be discussed below. It is known that when samples contain high enough defect concentration, all positrons in the samples should be trapped by defects and annihilate in defects; thus, the bulk lifetime component can be neglected. Referring to the literature (Brandt and Dupasquier 1983; Nielsen *et al.* 1982) for assignment of lifetime components in the present work, the shorter lifetime τ_1 (187 to 236 ps) may be regarded as the average value of positron lifetimes in vacancies and dislocations, and the longer lifetime τ_2 (368 to 558 ps) may be associated with positrons annihilating in voids. Considering that the positron lifetime for dislocations is shorter than it is for vacancies (Mackenzie 1983), the variations of τ_1, τ_2, I_1 and I_2 with increasing N can be explained as follows. In stage B, the dislocation density is decreased, so an increase of τ_1 accompanied by an increase of I_1 indicates that the vacancy concentration in the samples is rapidly increased and the total concentration of dislocations and vacancies is also increased. This case is possible, because in stage B the dynamic recovery makes the fixed dislocations move anew and cancel each other out, so it not only decreases the density of dislocations but also produces a large amount of vacancies. In stage B voids grow and become gradually micro-cracks, contributing to an increase of τ_2 and a decrease of I_2. In stage A, because there is no dynamic recovery, the vacancy concentration will rapidly fall and the density of dislocations produced by microdeformation is increased. The total concentration of dislocations and vacancies is decreased, and thus τ_1 and I_1 decrease. In stage A the decrease of τ_2 and increase of I_2 may be attributed to stopping of void growth and clustering anew of vacancies. As mentioned above, I_1 and I_2 vary inversely due to them having a constant sum, and thus the variation of one can introduce an inverse variation in the other.

4. Conclusions

The microhardness measurement can be used to probe fatigue hardening and fatigue softening in the process of thermal fatigue and PALS is a useful method to probe thermal fatigued defects in complex alloys. The variations of the positron lifetime parameters with N in the thermal fatigued alloys are different from those in mechanical fatigued metals and alloys. Here the positron lifetime parameters with N exhibit quasi-periodic up-and-down variation, but in mechanical fatigue they increase monotonically with N. In the thermal fatigued samples, it is proposed that the variations of positron lifetime parameters with N result mainly from variations of dislocation density and vacancy concentration caused by microdeformation and dynamic recovery dominating alternately. These two effects are also causes of fatigue hardening and fatigue softening.

References

Brandt, W., and Dupasquier, A. (Eds) (1983). 'Positron Solid State Physics' (North Holland: Amsterdam).

Grobstein, T. L., Welsch, G. E., Panigrahi, N., and McGervey, J. D. (1985). *In* 'Positron Annihilation' (Eds P. C. Jain *et al.*), pp 573–5 (World Scientific: Singapore).

Hautojarvi, H. (Ed.) (1979). 'Positrons in Solids' (Springer: Berlin).

Karjalainen, L. P., Judiu, T., and Karras, M. (1982). *In* 'Positron Annihilation' (Eds P. G. Coleman *et al.*), pp. 461–3 (North Holland: Amsterdam).

Mackenzie, K. (1983). *In* 'Positron Solid-State Physics' (Eds W. Brandt and A. Dupasquier), pp. 169–264 (North Holland: Amsterdam).
Maim, S., and Norstrom, L. A. (1979). *Metal. Sci.* **9**, 544.
Nielsen, B., van Veen, A., and Caspers, L. M. (1982). *In* 'Positron Annihilation' (Eds P. G. Coleman *et al.*), pp. 441–2 (North Holland: Amsterdam).
Tang, C. Q., Shi, Z. H., and Li, X. Z. (1993*a*). *Phys. Stat. Sol. (a)* **137**, K5.
Tang, C. Q., Li, X. Z., and Shi, Z. H. (1993*b*). *Cryst. Res. Technol.* **28**, 531.
Tang, C. Q., Li, G.. Y., Shi, Z. H., Li, X. Z., and Yao, B. T. (1993*c*). *Int. J. Fatigue* **15**, 515.

Manuscript received 8 May 2000, accepted 12 January 2001