

Strain determination in a friction-welded sample using non-destructive neutron diffraction.

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Abstract

Neutrons can penetrate thick layers of many engineering materials and therefore provide a useful technique to investigate internal strains reproduced by lattice deformations in a nondestructive way. The method uses high-resolution neutron diffractometry to determine lattice parameter variations $\frac{d-d_0}{d_0}$ of the order of 10^{-3} to 10^{-4} caused by internal or external forces. The strain distribution across the weld in an friction-welded sample of a Ni-base superalloy (*Waspaloy*) has been investigated. To determine the absolute amount of deformation, the unaffected lattice parameters d_0 must be known. The lattice parameters in regions far from both weld and heat affected zone seem to be a good choice, whereas a high-resolution measurement on *Waspaloy* powder clearly showed prestress of the powder grains; subsequent annealing caused the precipitation of a γ' -phase. The results of the experiments are presented and discussed and conclusions are drawn concerning the design of a neutron spectrometer optimized for strain measurements at Geesthacht.

1 Introduction

External forces as well certain methods of fabrication and processing cause deformation which can be observed microscopically as displacements of atoms from their original equilibrium positions. Elastic deformation under tensile forces tends to enlarge the spacings between lattice planes, whereas at the same time compressive forces act in the orthogonal direction. The well-known property of a three-dimensional solid-state "grid" to reproduce a diffraction pattern of x-rays or neutrons may thus be used to investigate this deformation. For metals, the absorption coefficient of x-rays is much larger than for neutrons, so that with x-rays surface strains can be investigated whereas neutrons in principle allow depth profiling of the bulk material. This paper discusses first results of neutron measurements on a Ni-base superalloy friction-welded sample. The experiments were made to study the advantages and disadvantages of the method and to establish design criteria for a neutron diffractometer optimized for strain measurements.

2 Neutron diffraction strain measurements in bulk material

The well-known BRAGG's law $= 2 d_{hkl} \sin\theta_{hkl}$ is the physical basis for all diffraction methods of strain determination. λ is the wavelength of either the electromagnetic or the particle wave, diffracted via the scattering angle $2 \theta_{hkl}$ from lattice planes $\{hkl\}$, with interplanar spacings d_{hkl} from each other. Whereas the electromagnetic wave is scattered by the electron cloud, the neutron is scattered predominantly by the nucleus in the center of this cloud. These different interaction mechanisms have important consequences: Since the nucleus acts as a point-like scattering center, neutron scattering is isotropic and the scattering amplitude does not depend on the scattering angle, as for x-rays. A second, even more important consequence is, that the absorption of x-rays is directly related to the electron density of a material, whereas for neutrons - which have no electrical charge, but a magnetic moment - the nuclear absorption and scattering properties of that material prevail. The intensity of x-rays for most engineering materials is reduced by a factor of 2 by layers of less than $50 \mu\text{m}$. Strain fields determined from x-ray measurements are considered to be two-dimensional. Neutrons on the other hand are capable of penetrating material layers which are three orders of magnitude thicker, so that three-dimensional investigations can be made and complete strain tensors can be determined immediately. In the following table neutron absorption for some selected Ni-base alloys has been calculated. Comparison is made to stainless steel and to the very strong neutron absorbers Gadolinium and Cadmium, which may be used to set up apertures that allow the precise definition of volume elements in a sample.

Neutron spectrometers used for strain measurements have to have an extremely good instrumental resolution, since typical lattice strains $e = \frac{(d-d_0)}{d_0}$ of the order of 10^{-3} to 10^{-4} . General purpose powder diffractometers (like D1A at the ILL-Grenoble, GPPD

material	density ρ [g/cm ³]	total macroscopic cross section Σ [cm ⁻¹]	neutron absorption thickness [mm]	
			$t_{.5}$	$t_{.1}$
Ni80/Cr20	8.40	2.20	3.16	10.5
Astroloy LC	8.00	2.14	3.25	10.8
Monel 400	8.84	2.12	3.27	10.9
Inconel 600	8.42	2.07	3.35	11.1
Inconel X	8.25	2.06	3.36	11.2
Hasteloy 'B'	9.24	1.98	3.51	11.6
Hasteloy 'C'	8.94	1.86	3.73	12.4
Inconel 718	8.19	1.71	4.05	13.5
Waspaloy	8.25	1.68	4.14	13.7
stainless steel type 304	7.93	1.17	5.91	19.7
gadolinium	7.95	1.49×10^3	0.46×10^{-3}	1.54×10^{-2}
cadmium	8.65	1.17×10^2	5.90×10^{-2}	0.196

(microscopic cross sections for thermal neutrons ($\lambda = 1.8 \text{ \AA}$) were taken from Ref. 1)

Table 1: Neutron absorption properties of some Nickel-base alloys.

at ANL-Argonne, NPD at LANSCE-Los Alamos, BSS in Harwell or MINI-SFINKS in Leningrad) all have $\Delta d/d_0$ resolutions of $1 \dots 3 \times 10^{-3}$, so that the required accuracy must be reached by good statistics. Under the assumption of a gaussian-shaped diffraction peak assuming 99.9% confidence this means a minimum of $n = 1.1 \times 10^5 \times \sigma^2$ counts per peak, or 10000 counts per peak for a typical standard deviation of 0.3° . This can only be achieved within a reasonable measuring time with a high-intensity neutron source, or an instrument which uses the steady state neutron flux from a reactor in the most efficient way. Neutron time-of-flight spectroscopy in this respect has certain advantages over crystal diffractometry, since the whole energy spectrum of the neutron source may be used to investigate many diffraction peaks simultaneously; the strain information comes from crystallites contained in a well-defined volume, and neutron detectors may look from many directions into the specimen either for strain depth profiling or simultaneous determination of the six components necessary to establish the strain tensor. Details of the neutron diffraction method and results that show its possibilities can be found in the excellent review article by Allen, Hutchings, Windsor and Andreani [2].

3 Strain distribution in a friction-welded sample

It is known that welding is one of the more important causes of residual stresses in materials. A friction-welded tube was chosen as a sample because this welding method can well be expected to leave internal strains in the material and there will be no contamination from other materials in the welding seam. Friction-welding is the preferred method for joining bars or tubes when a very narrow extension of the welding zone must be guaranteed in order to avoid brittle intermetallic phases. The welding times are extremely short and the temperature-cycle is superimposed by an intensive plastic deformation, which removes the intermetallic phases from the binding zone, whose structure at the same time experiences a strong refinement of the grains (cf. [3]). This grain refinement can be seen in Fig. 1.

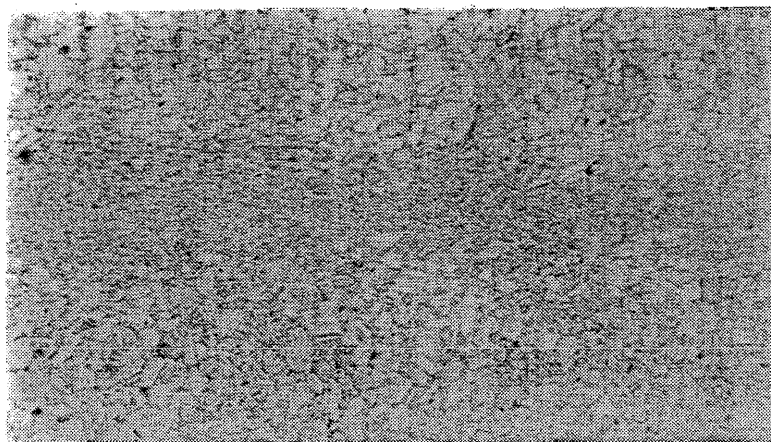


Figure 1: Photomicrograph of the welding zone of the friction-welded *Waspaloy* sample.

A sample of the Ni-base superalloy *Waspaloy* was manufactured by MTU-München. For the test specimen two tubes (48 mm outer diameter and 14 mm inner diameter) of the same material were welded. The strain distribution across the weld was scanned with a neutron beam of $4.5 \times 13 \text{ mm}^2$ in overlapping steps of 2 mm width at the D1A spectrometer in Grenoble. Four diffraction peaks ((111), (200), (220) and (311)) were investigated. For all reflections the scattering vector pointed essentially in the radial direction. The observed positive strains, plotted in Fig. 2, are therefore due to radial tensions in the material which increase towards the weld.

If a Young's modulus of 209 GPa is assumed for *Waspaloy* at room temperature, a strain of 1.5×10^{-3} results in a stress of 320 MPa, which is about 35% of the yield stress of the material. The strains were calculated with reference to the lattice spacings measured far from the heat-affected zone, which were considered to be strain-free. The fact that under this assumption only positive strains (*tensile forces*) can be observed, led us to an additional measurement on *Waspaloy* powder, in order to arrive at better lattice spacing

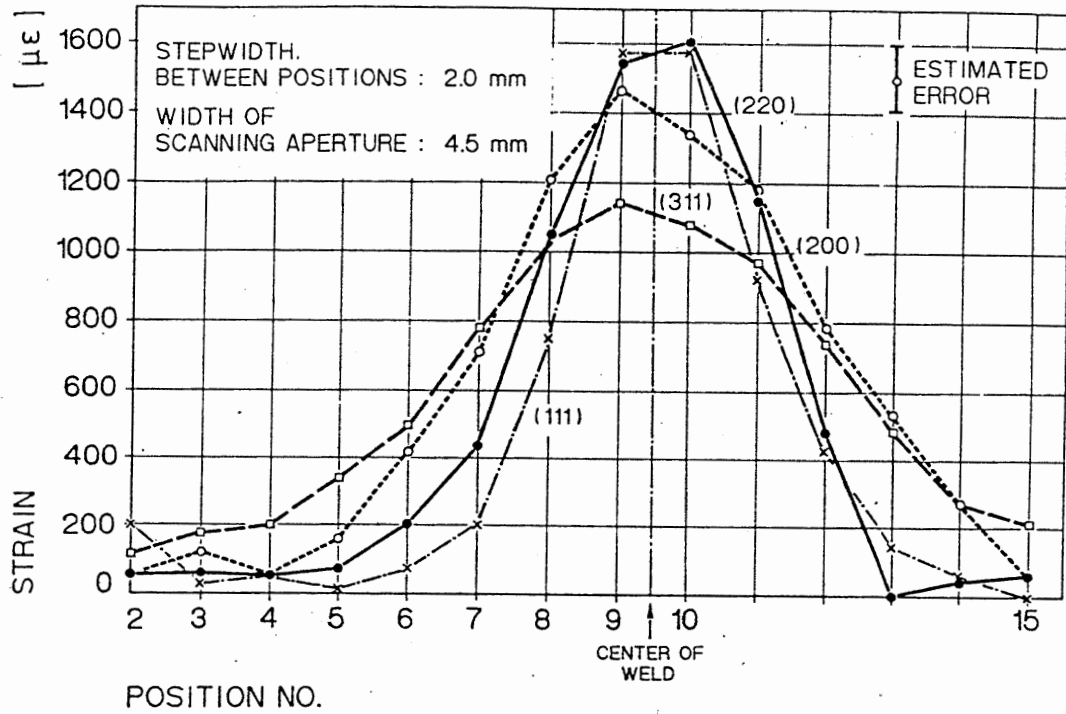


Figure 2: Strain distribution across the weld for different diffraction peaks.

reference values. According to the manufacturer, this powder was produced by spraying and rapid cooling, so that internal stresses were to be expected. Two measurements have been made, one using the *Waspaloy* powder as received and one after annealing it for 1 hour at 900 °C. The experiments were carried out on a high-resolution neutron time-of-flight spectrometer. The results are shown in Fig. 3.

Each diffraction peak in Part A of Fig. 3 is shifted towards a smaller d -value and accompanied by a smaller satellite peak in Part B after annealing. We interpret this as the appearance of an intermetallic γ' -phase in the thermally treated material. As an example the detailed structure of the most prominent (311)-peak is shown in Fig. 4.

The (311)-lattice spacing for the as-received powder is $d = 1.0842 \text{ \AA}$. This value is shifted after thermal treatment to $d = 1.0778 \text{ \AA}$ and a satellite appears at $d = 1.0698 \text{ \AA}$. The peak shift is due to the release of internal strains. At the same time peak broadening is observed: After fitting the peaks with gaussians, we find

$$\begin{aligned}\sigma &= 0.0026 \text{ \AA} \text{ for the as received powder} \\ \sigma &= 0.0064 \text{ \AA} \text{ after thermal treatment and} \\ \sigma &= 0.0117 \text{ \AA} \text{ for the satellite peak}\end{aligned}$$

These experimental findings are not yet fully understood, but it must be emphasized that special care has to be taken in determining the reference lattice spacings, if absolute strain values are to be found.

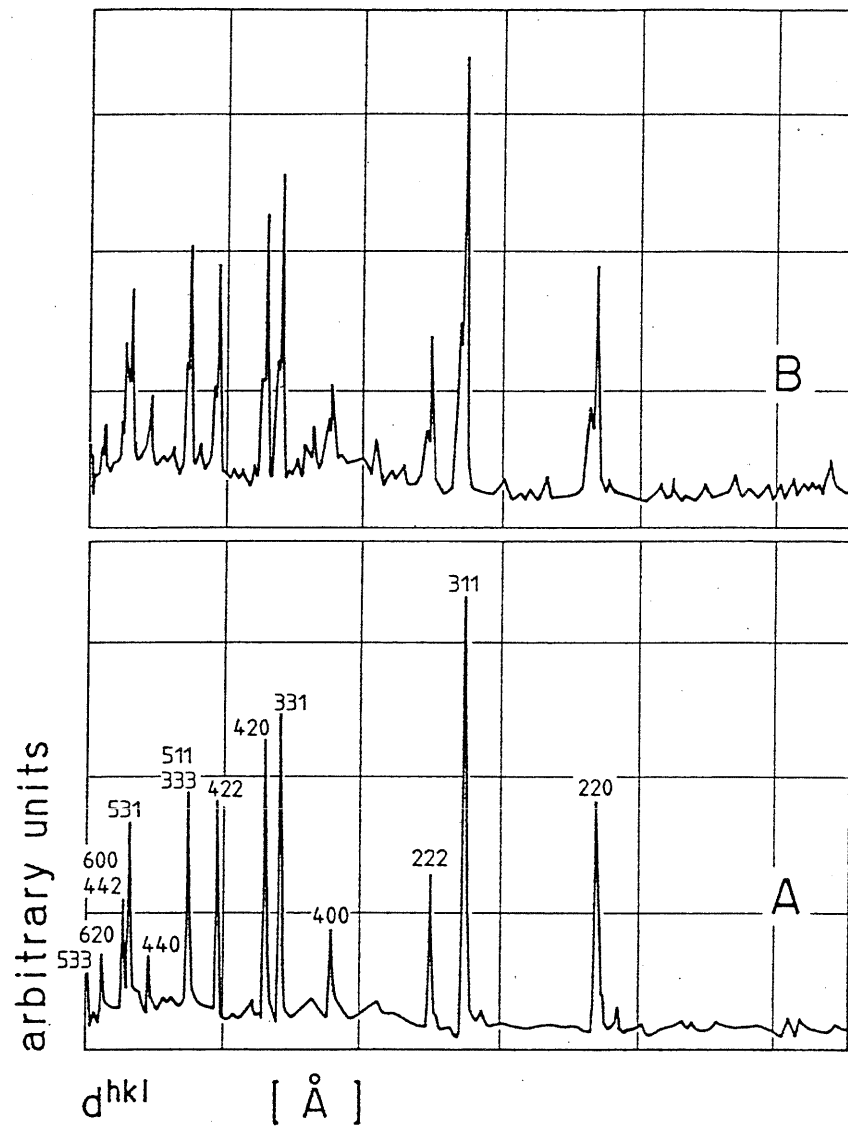


Figure 3: High-resolution powder diffractograms of *Waspaloy* (A: as received, B: annealed for 1 h at 900 °C).

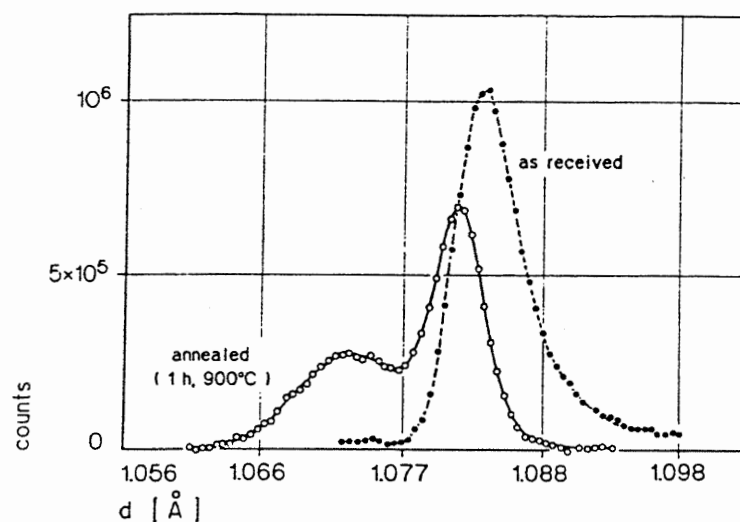


Figure 4: Detailed structure of the (311)-peak.

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