METHOD FOR *IN SITU* OBSERVATION OF STRESS-INDUCED MARTENSITE FORMATION AND EVOLUTION IN SHAPE MEMORY ALLOYS

BY

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Abstract The recent development of shape memory alloys (SMAs) led to the conclusion that no shape memory effect (SME) exists unless a "cold shape" (characteristic to martensitic domain) different to initial "hot shape" (characteristic to austenitic domain) can be induced to a SMA specimen. The microstructural phase that accompanies cold shape obtainment is termed stress-induced martensite (SIM). To observe SIM during in situ experiments, special devices are necessary in order to keep the specimens in deformed state. The present paper describes an original method for *in situ* observation of SIM by means of two experimental devices. The former is adapted to optical microscopes table. It is applicable for lamellar specimens which are elongated while being examined under microscope. This allows good *in situ* observation of SIM formation. The latter is used in conjunction with a tensile testing machine and is meant to "freeze" elongated specimens in tensioned state, in order to be analyzed by X-ray diffraction. The structure and functionality of the two devices are introduced in the paper together with corresponding examples of *in situ* observation of SIM formation and evolution with increasing applied stress.

Keywords: shape memory alloys, stress-induced martensite, microstructure, optical microscopy, X-ray diffraction

2000 Mathematics Subject Classification: 53B25, 53C15

1. Introduction

The pseudoelastic behaviour is associated with mechanical memory, defines any non-linearity occurring on the unloading portion of a stress-strain curve. In the case of classical materials, unloading portion is parallel with loading elastic portion (BC // 0A), as illustrated with solid line in Fig. 1.

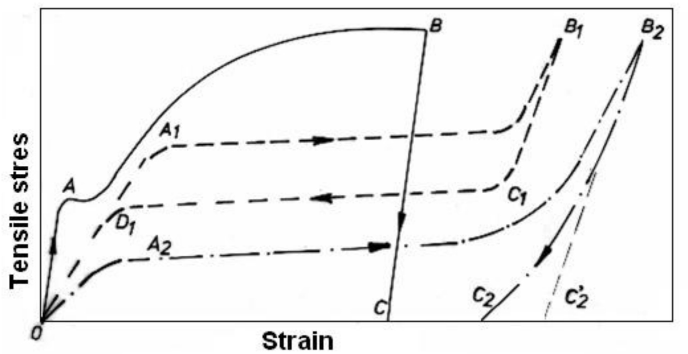

Fig.1 Schematic stress-strain curves comparing classical tensile behaviour (solid line) with reversible stress-induced martensitic transformation (dashed line) and crystallographic reorientation-induced martensitic transformation [1]

Superelasticity ($0A_1B_1C_1D_10$) and crystallographic reorientation ($0A_2B_2C_2$) are characterized by loading plateaus along which stress induced martensite (SIM) is reversibly and irreversibly formed, respectively. Basically superelasticiy occurs within the thermal range $A_f < T < M_d$, where M_d is the maximum temperature for SIM formation (above M_d diffusion intervenes) [2].

The present paper aims to introduce a method for *in situ* observation at room temperature (RT) of SIM formation and evolution in shape memory alloys (SMAs) by means of two special experimental devices.

2. Experimental Devices

For the study of SIM formation, by means of optical microscopy (OM), the device shown in Fig.2 was designed and manufactured, in order to be used with the microscopes *IOR MC9* and *KARL ZEISS JENNA*. The execution drawing of the device is shown in Fig. 3. Observations were performed at room temperature with high-aperture objective lens, by continuously observing a 4 square millimetres surface along the deformation direction. The specimens are band-like with the dimensions 100 x 4 x $0.5\cdot10^{-6}$ m and have a narrow section on the active gauge, as illustrated in the detail from Fig. 3. After being ground, and polished, the specimens were fastened in the mobile grips (2) and the fix grips (3) by means of the screws (4), according to Fig.3. On the movement screw (7) a dynamometric key is fastened, able to measure torques up to 50 N·m. After installing the device on the optical microscope, the specimen is strained by means of the dynamometric key. The micrometric drummer (6) enable determining specimen strain with an accuracy of 10^{-4} m. Specimen strain relieving can be done by means of the dynamometric key or the lever (5).

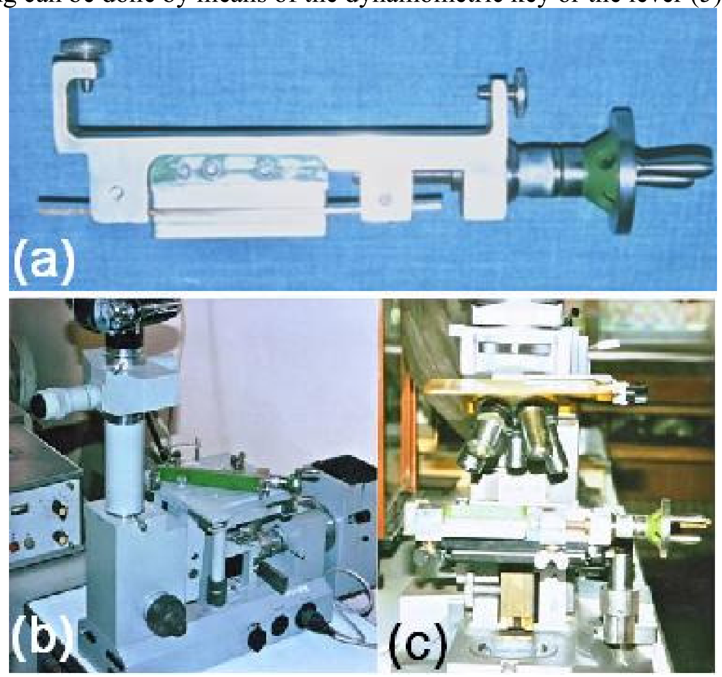

Fig.2 Experimental device for *in situ* OM observation of stress-induced martensite: (a) main view; (b) view of the device installed on a KARL ZEISS JENNA optical microscope equipped with EXACTA VAREX photo camera; (c) the device installed on a IOR MC9 optical microscope

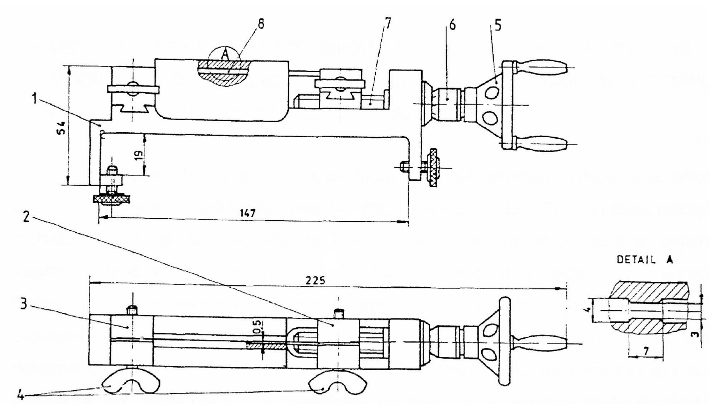

Fig. 3 Execution drawing of the device for in situ OM observation of SIM formation: 1-frame; 2-mobile grips; 3-fix grips; 4-fixing screws; 5-movement screw lever; 6-micrometric drummer; 7-movement screw; 8-SMA specimen

For determining the stress applied to the specimen relationship (1) is used:

$$\sigma \cong \frac{F}{S_0} \cong \frac{M_t}{d_2/2 \cdot S_0 \cdot tan\left[arctan(P/\pi d_2)+arctan\,\mu\right]} \qquad (1)$$

where M_t is the torque determined by dynamometric key, $S_0 = b_0 \cdot g_0$ is initial transversal cross-section of the specimen, d_2 – average diameter of the screw and μ – friction coefficient of screw-nut coupling

For the study of SIM formation by means of X-ray diffraction (XRD) a special straining-fastening device was designed and manufactured. The device is adapted for being used in conjunction with a FPZ 100/1 tensile testing machine and a DRON 2,0 X-ray diffractometer, as shown in Fig. 4.

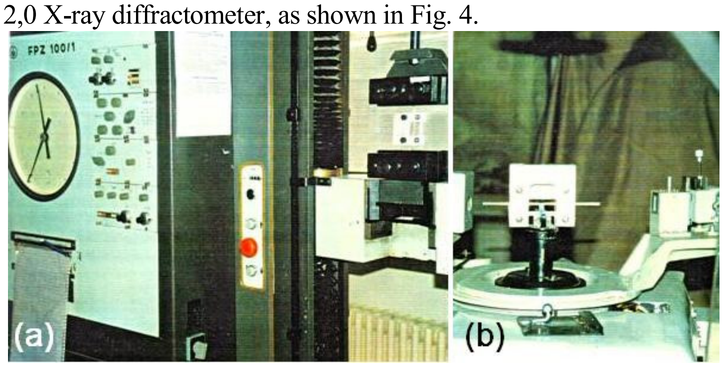

Fig. 4 Experimental straining-fastening device of lamellar specimens for *in situ* XRD study of SIM formation: (a) in fastening position of the specimen, on a FPZ 100/1 tensile testing machine; (b) in working position on a DRON 2,0 X-ray diffractometer

The device uses specimens with larger width as compared to the device for OM observation. The specimens, with dimension 2 x 20 $100 \cdot 10^{-3}$ m, are elongated by means of a tensile testing machine, as in Fig. 4(a) up to the desired stress. Then the device blocks the elongated specimen in strained state and enables its transportation to the diffractometer, where XRD profiles are recorded at RT, Fig. 4(b).

3. Experimental Applications

The results obtained by means of the two experimental devices are shown in Figs. 5 and 6 which illustrate in situ formation of SIM by OM micrographs and XRD profiles.

Fig. 5 reveals *in situ* formation of acicular SIM, with parallel orientation, at the stress of 20 MPa, no major changes being noticeable at 40 MPa.

Fig. 6 illustrates the occurrence of MIT by means of X-ray diffractograms and its aspect, by means of OM micrographs. On the diffractograms, the simple hatched areas correspond to the SIM peaks and the double hatched areas to the phases that totally or partially disappeared on loading.

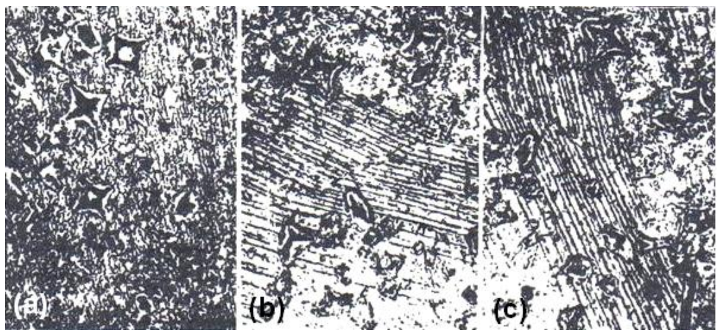

Fig. 5 *In situ* formation at RT of acicular SIM in Cu-Al-Ni SMAs: (a) initial state; (b) under a 20 MPa applied stress; (c) under a 40 MPa applied stress

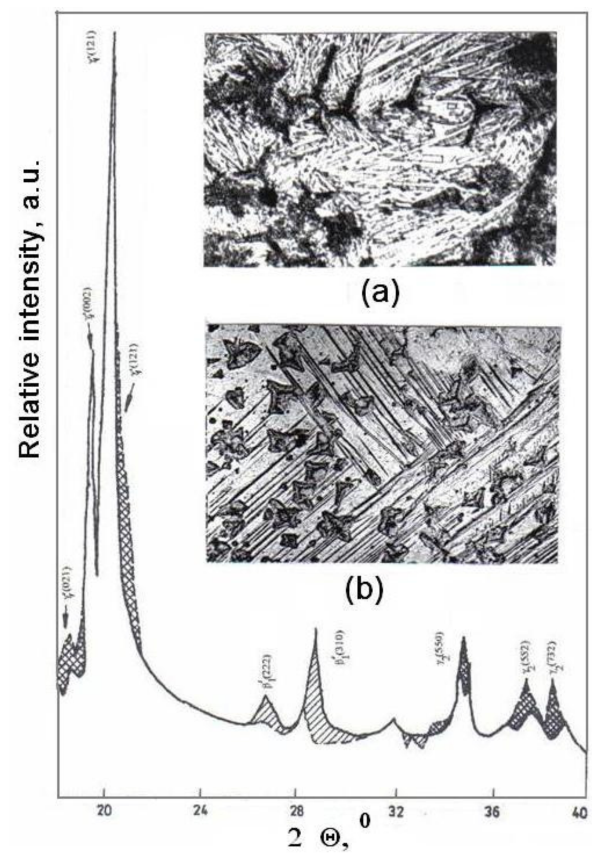

Fig. 6 Typical simplified diffractograms of initial state (dash line) and of elongated state under a stress of 100 MPa (solid line)as well as microstructures illustrating *in situ* effects of SIM formation in a Cu-Al-Ni SMA. : (a) optical micrograph of initial state; (b) optical micrograph of the state stressed with 20 MPa

4. Conclusions

1. Two experimental devices for *in situ* observation of stress-induced martensite formation, in SMAs were introduced.
2. Two examples were presented of the study of stress-induced martensite formation in the specific case of a Cu-Al-Ni SMA, illustrating OM micrographs and XRD profiles recorded using the two experimental devices.

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METODĂ PENTRU STUDIUL *IN SITU* AL FORMĂRII ŞI EVOLUŢIEI MARTENSITEI INDUSE PRIN TENSIUNE ÎN ALIAJELE CU MEMORIA FORMEI

(Rezumat)

Dezvoltarea recentă a aliajelor cu memoria formei (AMF) a dus la concluzia că nu se poate obţine efect simplu de memoria formei (EMF) dacă unei probe din AMF nu i se poate induce o "formă rece" (caracteristică domeniului martensitic) diferită de "forma caldă" iniţială (caracteristică domeniului martensitic). Faza microstructurală care însoţeşte obţinerea formei reci este numită martensită indusă orin tensiune (MIT). Pentru a observa MIT, în timpul experimentelor *in situ*, sunt necesare dispozitive speciale pentru a păstra probele în stare deformată. Lucrarea de faţă descrie o metodă orginală de observare *in situ* a MIT, prin intermediul a două dispozitive experimentale. Primul este adaptat la masa microscoapelor optice. Este aplicabil pentru probele lamelare care sunt alungite în timp ce sunt examinate sub microscop. Acest lucru permite o bună observare *in situ* a formării MIT. Cel de-al doilea este utilizat împreună cu o maşină de încercat la tracţiune şi are rolul de a „îngheţa" proble alungite în stare tensionată, pentru a fi analizate prin difracţie de raze X. Lucrarea descrie structura şi funcţionalitatea celor două dispozitive, împreună cu exemple corespunzătoare de observare *in situ* a formării şi evoluţiei MIT la creşterea tensiunii aplicate.