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MICROSTRUCTURAL CHANGES IN STEP-WISE THERMALLY STIMULATED MARTENSITIC TRANSFORMATIONS

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Abstract- The "Step-wise stimulated Martensite to Austenite Reversible Transformation"(SMART) in NiTi alloys is here examined by means of X ray diffraction spectra detected:1) before the procedure required to activate SMART; 2) after each step of the procedure;3) after SMART. Results obtained prove that some specific reflections are affected by the procedure, notably the ones which correspond to the orientations apt to store the highest transformation strains.

1.-Introduction

Several shape memory alloys as NiTi, CuAlZn, widely known for their macroscopic recovery properties have in recent years been reconsidered in connection with hysteresis effects related to partial cycling(1-3).
Though general thermodynamic continuous macroscopic models have been advanced to explain the hysteretic effects of partial cycling, they have till now been applied to the CuAlZn system(3).
Other experimental findings on the same system have been obtained performing partial cycling in presence in several cases of one single phase, martensite or parent phase(1,2).
In the above cited cases attention is mainly directed to dissipative energetic terms connected with interface motion. Generally the procedure adopted to perform a sequence of partial cycles gives rise to a pile up of defects(2) and consequently to an increase of irreversible processes with increasing the number of partial cycles.In our opinion all above cited findings are to be considered in the frame of cumulative damage as found in fatigue.
Another distinct class of "micromemory" effects is connected with the "Step-wise stimulated Martensite to Austenite Reversible Transformation"(SMART), in the temperature domain, previously put into evidence(4). A local reversible micromemory can be imprinted following a sequence of incomplete cycles on heating, freely selected with a decreasing rank inside the reversion temperature range As,Af, as explained in previous papers (4,5); the macroscopic reversibility of SMART has therein also been shown, at least at the light of macroscopic properties.
SMART has moreover a correspondent step-wise stimulated parent-phase martensite

transformation in the stress domain(6), where partial cycling on the "pseudoelastic plateau" can imprint "stimulated Stress Drops in P-phase"(SDIP) and consequently a mechanical memory, freely selected within σ_s(PM), σ_f(PM) range. The main features and correspondences between SMART, built up during the reverse transformation and SDIP, built up during the direct one, have already been compared and contrasted(5). Some hypotheses(5,6) have been advanced to explain experimental findings, but a comprehensive microstructural explanation is till now lacking. Investigations have therefore been undertaken by X ray diffraction to enlighten the key characters underlying SMART.

2.-Experimental

Nearly equiatomic NiTi polycrystals were cut from the same hot rolled slab as previously used.
The specimens here examined have been previously submitted to a solution treatment at 900°C(1h), followed by water quench. Afterwards, in order to exclude any side effect due to stabilization, they have been submitted to a treatment of 60 complete thermal cycles between martensite and parent phase.
X Ray Diffraction(XRD) spectra have been detected on a D500 PC Siemens equipment interfaced to an AT IBM personal computer. Software packages are provided to handle the instrumentation and to perform the data reduction. CuK_α radiation length has been adopted. A sample holder, equipped with a liquid N_2 cooling accessory and with heating resistance coils, allows to investigate all the temperatures within -150°C, +200°C.
"Incomplete Cycles on Heating"(ICH), as already explained(4), were performed on the specimens, preliminarly conditioned as above specified.
Complete XRD spectra were detected preliminarly in the B2 phase (T=+120°C) and in B19' phase (T=-150°C). Peak indexing is taken from (7). Afterwards attention is directed to the most intense reflection peaks of the monoclinic B19' phase at -150°C:
1) before the ICH procedure;
2) after each ICH;
3) after SMART.

3.-Results

In order to assess the experimental conditions required to correctly perform ICH's, the growth of intensity of a high temperature phase peak has been checked as a function of temperature, on heating from T=-150°C < Mf to T=+120°C > Af.
Fig.1 shows the sequence of the intensities of the $110+1\bar{1}0$ unresolved doublet of the R-phase, during its growth on heating from the martensite phase. The data clearly show the steady intensity increase with increasing temperature up to 50°C along with a little shift in angular set: when temperature is increased to 60°C the peak undergoes an angular shift due to the collapsing of the unresolved 110 doublet of the R-phase into the 110 reflection of the B2 phase, with an abrupt shift as expected from a first order transition.
Fig.2 shows the normalized ([Ipeak-Ipeak(min)]/[Ipeak(max)-Ipeak(min)]) peak values of the reflections given in Fig.1, independently upon their angular position, as a fun-

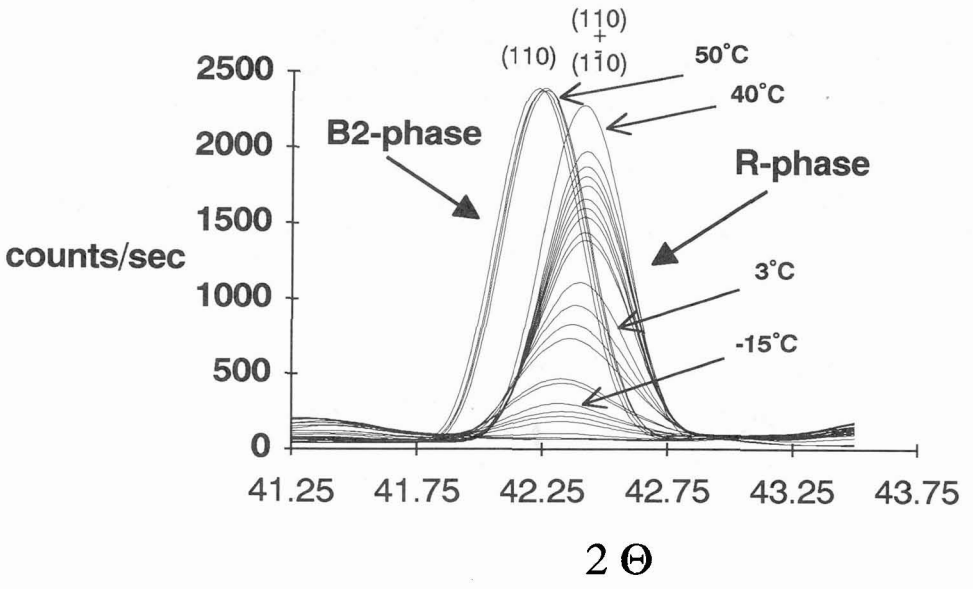

Fig.1 - Unresolved doublet (110)+(1$\bar{1}$0) of the R-phase during its growth on heating from martensite:the shift in angular position is due to the transformation in B2 phase.

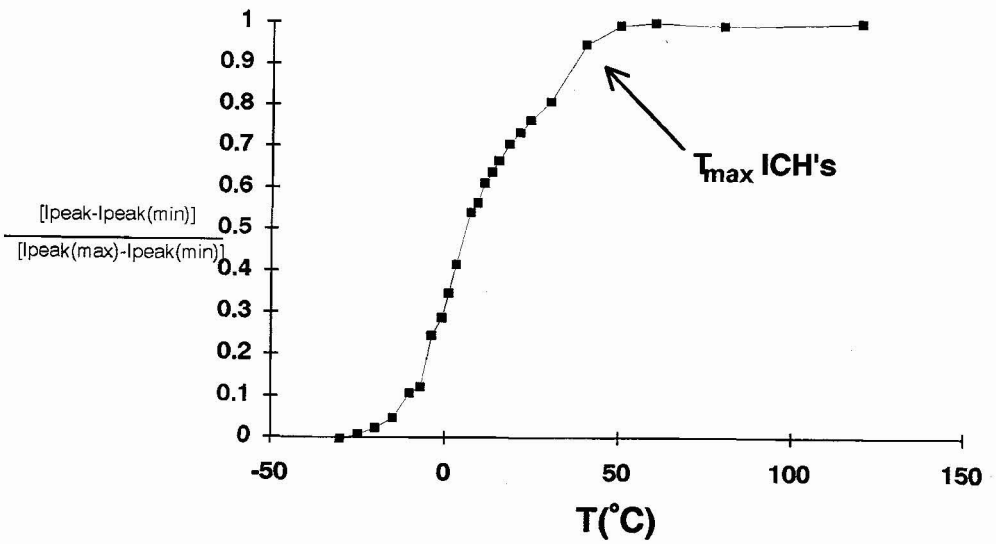

Fig.2 - Normalized peak values [Ipeak-Ipeak(min)]/[Ipeak(max)-Ipeak(min)] of the reflections given in fig.1.

ction of temperature: the data allowed to assess the sequence of temperatures for performing ICH.

The sequence of arrest temperatures, in decreasing order, ranked from 38°C, at 2°C step: 15 steps have been performed. This sequence has been adopted as a useful compromise between a lengthy procedure and a built in ordering sufficiently established to be detected.

Figs.3a and 3b show, respectively, the most intense diffraction peaks of the B19' phase (at -150°C), and an enlarged view of the unresolved (111)+(021) reflections of the same multiplet, before ICH procedure, after 15 ICH procedure and after SMART.

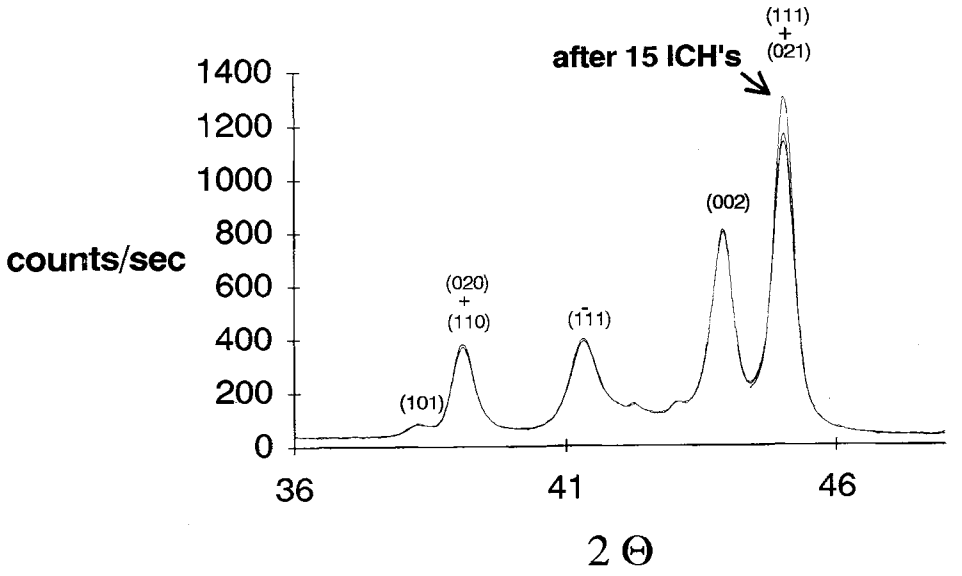

Fig.3a - The most intense reflections of the B19' phase at -150°C,before the ICH procedure, after 15 ICH and after SMART

Fig.4 shows both the decreasing sequence of ICH's stop temperature and the intensity percent increment of the B19' (111)+(021) unresolved reflections (taken at T=-150°C, after each ICH) as a function of ICH number.

4.-Discussion

As it can be seen some peaks appear more affected than others: notably the unresolved (111)+(021) appears sensibly affected by the ICH procedure. Some others, as for instance 002 reflection, appear slightly modified.

In any case however all peaks which are affected in intensity by ICH procedure, invariably regain, after SMART, approximately the same intensity value they had before the procedure to activate SMART. This is clearly evident in fig.3b and beyond proving the reversibility of the process, affords to exclude any side effect due to in-

complete stabilization. The reversibility of the process, already proved macroscopically (4,5), is now proved microscopically.

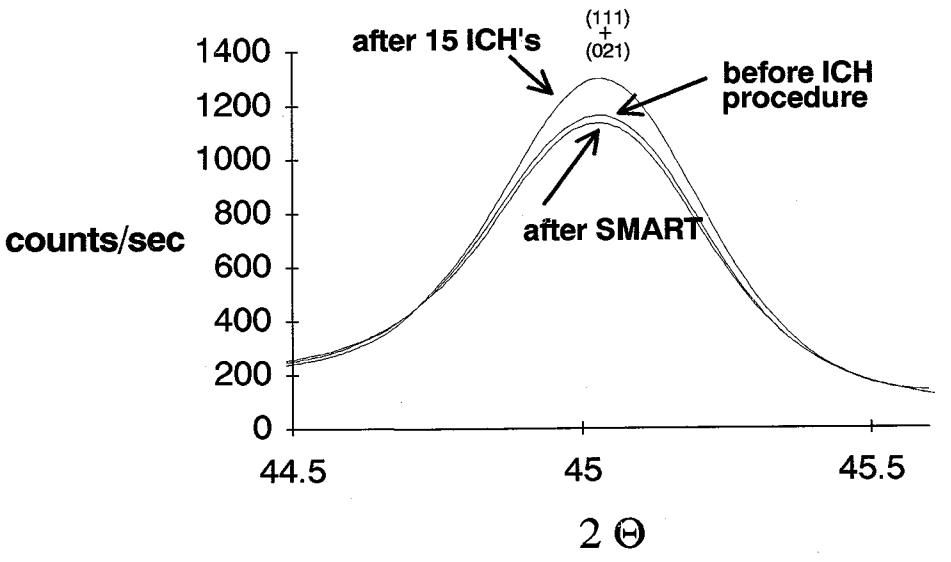

Fig.3b - Enlarged view of the (111)+(021) reflections given in fig.1.

As it can be appreciated from fig.3, the diffraction peaks most affected by ICH procedure, requested to activate SMART, appear to involve the same cristallographic directions along which it is easier to store transformation strain(8).

5.-Conclusions

SMART appears consequent to a procedure of ICH which imprints a number of "micromemories" steps, completely erasable by the SMART itself.
The reversibility character of the procedure adopted to imprint it, at the light of present results, is proved also from a microscopic point of view.
The diffraction peaks most affected by ICH procedure, requested to activate SMART, appear to involve the same cristallographic directions along which it is easier to store transformation strain.

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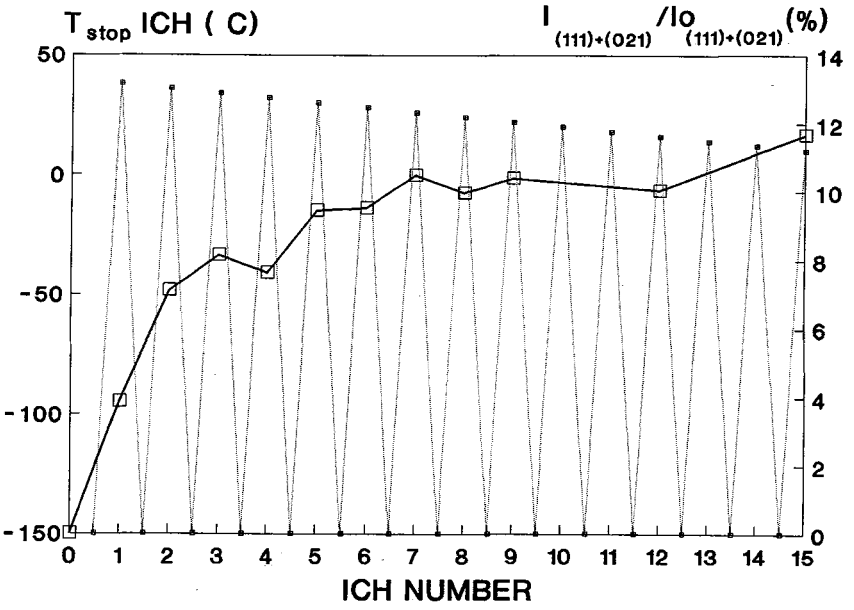

Fig.4 - Percent increment of intensity of the B19' (110)+(021) reflections as a function of ICH number.

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