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MECHANICAL PROPERTIES OF THE COLD-WORKED MARTENSITIC NiTi TYPE ALLOYS

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Abstract.- Recently, it has been shown that the cold-worked NiTi alloy has unusual pseudo-elastic properties in the martensite phase.In this communication further mechanical properties of NiTi and NiTiCu alloys in the cold-worked state are presented. It is shown that both have a higher elastic limit, a higher ultimate tensile stress and a different behaviour during the plastic deformation (i-e a large necking is observed) than the annealed alloys. These unusal mechanical properties have been explained in large part in terms of dislocation - dislocation and dislocation-martensite interface interactions. However some observed features are only understood if, in addition, the premartensitic phase (R-phase) is present and active during the phase transformation.

1. Introduction.- There has been considerable recent interest in the mechanical behaviour of shape memory alloys based on near equiatomic NiTi and (NiCu)Ti compositions. A review of data, measured on these alloys can be found in (1) and they can be summarised in the following way:

- in the martensite phase, a low yielding ($\sigma_{0.2}$) exists, which corresponds to the stress to reorient martensite by twinning or to assist growth of one martensite orientation at the expense of an adjacent, unfavourably oriented one.
- above M_s, an increase of $\sigma_{0.2}$ occurs, which is well explained by the stress induced martensite mechanism. The increase of $\sigma_{0.2}$ continues, until the yield stress σ_p for plastic deformation is more favorable. When both have about the same value, a minimum of ductility is observed, which is explained by the unfavorable effect of the simultaneous presence of conventional slip and of martensite growth, each restricting the motion of the other. σ_p was interpreted as the stress at which dislocation processes begin to occur on a large scale. All these measurements were done on well annealed specimens, this state being usually the one which gives the best shape memory effect. Therefore, it is very surprising that cold-worked (c-w) specimens show improved mechanical and shape memory effects. More on that is reported in this paper.

2. Experimental.- The NiTi based alloys were prepared using standart technique (2). The composition and the transformation temperature M_s of the different alloys are given in Table . After the thermo-mechanical treatment, tensile and damping specimens were machined directly out of the cold-deformed rods. The amount of cold-drawing done after the last heat treatment is given in relative area reduction. Tensile specimens with thread, 3 mm in diameter with a gage length of 17.5 mm were used for the cold-worked material, whereas specimens without thread, 5 mm in diameter with a gage length of 25 mm, were used for the well annealed material (Fig.1a). The dynamic Young's modulus and the damping were measured during thermal cycling between 100 and 400 K on plate of 50 mm x 5 mm x 1 mm in using a resonant bar apparatus (3) working in the kHz range. The value of the Young's modulus was used to

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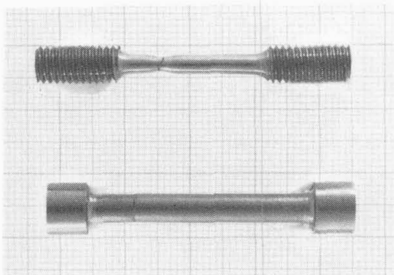

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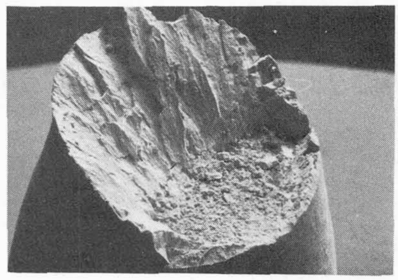

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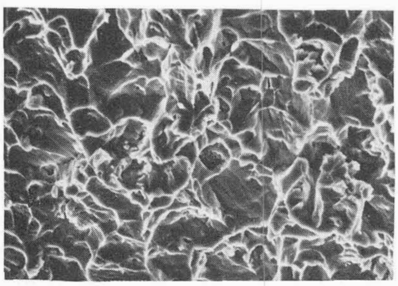

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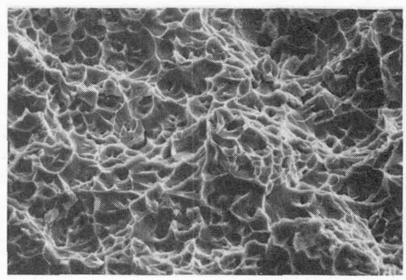

Fig. 1: Photos of the fractured tensile specimens 1 and 4 and of the corresponding fracture surface:

a) above the c-w (1) below the annealed specimen (4). The grid is metric with a mesh of 1 mm
b) fracture surface of 1 (x 12)
c) detail of the fracture surface of 4 (x 480)
d) detail of the fracture surface of 1 (x 480)

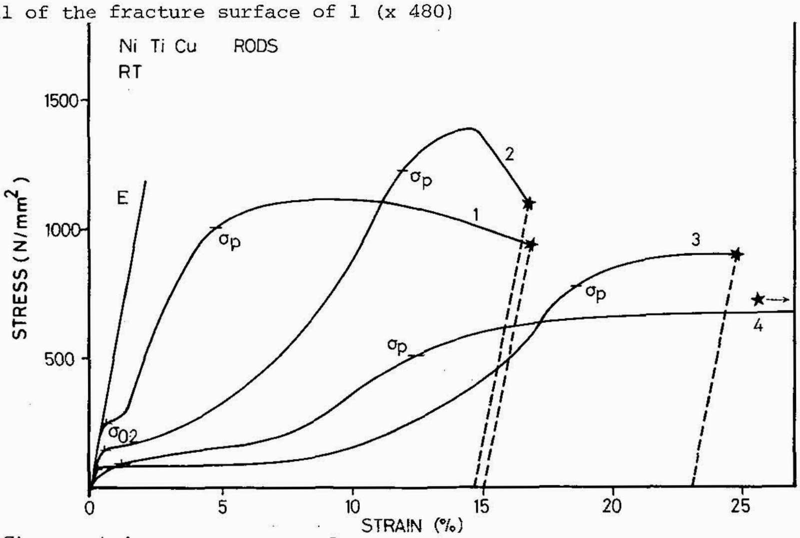

Fig. 2: Stress-stain curves measured at room temperature. Curve 1: c-w specimen, curve 2: specimen c-w and annealed at 300 C; curve 3: specimen c-w and annealed at 500 C; curve 4: annealed specimen (950 C).

correct the stress-strain curves form the effects of the deformation of the tensile machine. For the wires, no correction was done.

3. Results and discussion.- Stress-strain curves of alloy NiTiCu in different states, defined in Table , are drawn on Fig. 2. Photos of the fracture surface of specimens 1 and 4 are shown in Fig. 1. These 4 specimens were measured at room temperature, below M_s. One observes that the yield stress $\sigma_{0.2}$ is the highest for 1 (3 times higher than 3); then its value decreases with the anneals performed at relative low temperatures (2 and 3). The plateau, due to the martensite reorientation is almost nonexistent on the curve 1 and it is the largest on curve 3, where it is even larger than the one of the well annealed specimen. This plateau is followed by a new increase of stress, which is usually explained by the elastic deformation of existant martensitic plates, by the crossing of martensite plates, by the formation of other types of martensite and by some early plastic deformation (4). However, for the c-w specimens the strain associated with this increase of stress recovers when the load is removed; this corresponds to the pseudo-elastic behaviour found on cold-worked wires, as explained further in the text. This stress increase is followed by the real plastic deformation stage, which begins at σ_p. It is the highest for 2 (1200 N/mm^2), which is about 2.4 times higher than the σ_p of 4. For specimens 1 and 2, the plastic deformation is caracterised by a large necking with a surface reduction of 30 to 40 % (Fig. 1b shows an example). In contrast specimen 4 deforms homogeneously, without necking (Fig. 1a) until rupture, after about 40 % elongation. A detailed observation of the 2 fracture surfaces (Fig. 1c and 1d) shows that they are not very different, even if a finer structure is apparent for the c-w specimen.
Above A_s, it is known that $\sigma_{0.2}$ increases with increasing measuring temperature. This is also observed on the c-w specimens for both NiTi and NiTiCu alloys (Fig. 3). As expected $\sigma_{0.2}$ increases until its value is equal to σ_p. On this figure, the values of UTS are in true stress, calculated at the rupture surface. One observes that σ_p (between 1000 and 1300 N/mm^2) and UTS (between 1300 and 1600 N/mm^2) lie in a relative largs scatter band and that no clear tendency of increase or decrease of them is observed. Fig. 4 shows the pseudo-elastic behaviour of a NiTiCu specimen measured at 100 C. One observes a strain recover larger than 8 %, when the deformation stress of 1150 N/mm^2 is released. The stress-strain curve shows also a kind of upper yield point, followed by a slight decrease of stress and a plateau, due to the stress-induced martensite. The E modulus, as well as the damping Q^{-1} were measured on specimen 1, 3 and 4. Measurements made during cooling are reported into Fig. 6 . One observes above M_s a large decrease of E, which is the largest for specimen 1; simultaneously, an increase of Q^{-1} by a factor $\sim$ 100 is present; curve 1 shows the highest damping above M_s. In the martensite phase the E modulus have the same slope, but different absolute values; for instance E of 1 is about 30 KN/mm^2 (or 60 % to 30 %) lower than E of 4. The damping of specimen 1 and 3 barely increases in the martensic phase, whereas a nice maximum, located below M_F at 10 C is observed for the well annealed specimen. At last, Fig. 5 shows the stress-strain curves of 2 NiTiCu wires, cold-drawn at different amount (a at 80 %, b at 45 %). The stress-strain curve of a follows the straight line of E almost until rupture, which occurs after 4 % deformation at a stress of 1800 N/mm^2. The curve b has a slower increase of stress with a certain curvature. After 20 % deformation, the stress of b is 70 % of the one of a. Rupture of b occurs at 1300 N/mm^2 after 6 % deformation. Both wires show a pseudo-elastic behaviour almost until rupture.

From all these measurements, one concludes that the general behaviour of the cold-worked specimens are not very different than the one of the well annealed specimens, except that all stresses are much higher. In addition, for the c-w specimens, the stable reorientation of martensite is partially or completely prevented below M_s; this effect is the most pronounced for the wires and for specimens, which were not annealed above A_F before the measurement; a pseudo-elastic behaviour is then ob-

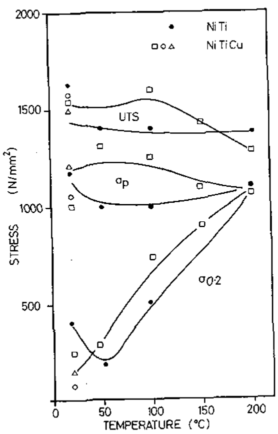

Fig. 3: Yield stress $\sigma_{0.2}$, plastic stress σ_p and UTS for NiTi and NiTiCu as a function of temperature.

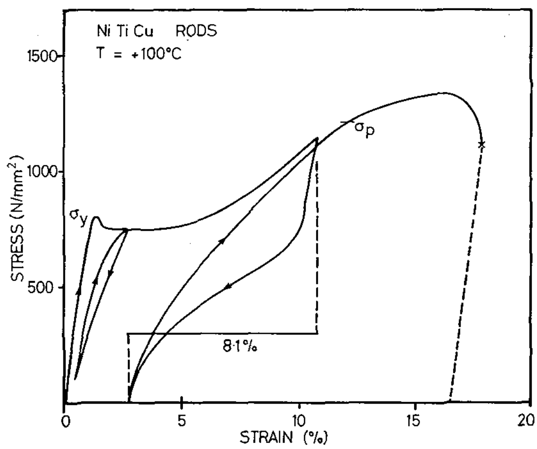

Fig. 4: Stress-strain curve of a NiTiCu specimen measured at 100 C.

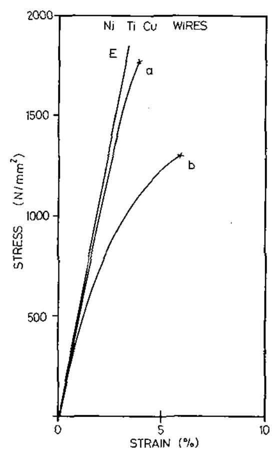

Fig. 5: Stress-strain curves of 2 wires (ø 0.5 mm) 80 % c-w a and 45 % c-w b.

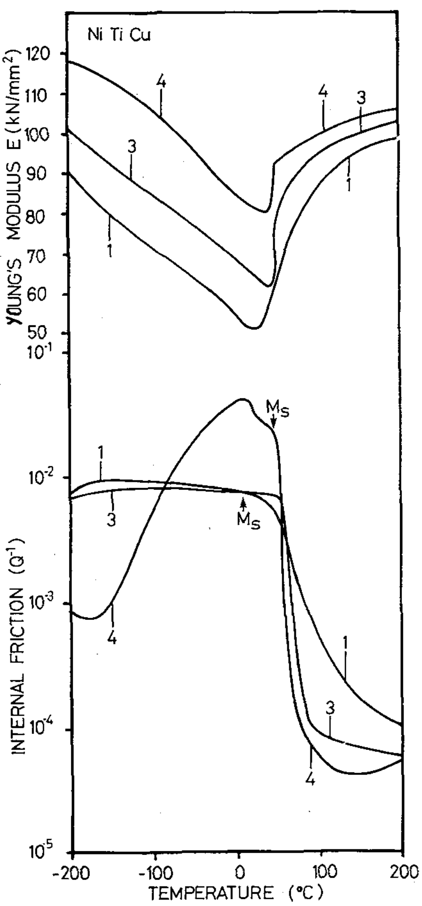

Fig. 6: Young's modulus E and damping Q^{-1} of a specimen in the cold-worked state (1), annealed at 600 C (3) and 900 C (4).

served. At all temperatures, between RT and 150 C, the plastic flow stress has about the same value and the plastic deformation is non homogenous, with a large necking. For specimen cold worked and annealed at 500 C, a large plateau with no stress increase is observed at RT; other measurements on this specimen have shown that large 1-way effect (→ 11.4 %) and 2-way effect (→ 2.5 %) are observed.

Tadaki and Wayman (5) observed on cold-drawn specimens a fine lamellar martensite, with immobile structure and explained the pseudo-elastic effect observed below M_s by microtwin appearance under loading, which are then unstable when the load is removed. Similarly, most of our observed effects can be explained by the presence of a large quantity of dislocations, which prevent partly or completely the stable reorientation or the growth of the martensite plates. These dislocations pin also the martensite interfaces, which explains the relative low Q^{-1} in the martensite phase of the c-w specimens; these dislocations are also a barrier to the motion of dislocations, responsible of the plastic flow. During anneals at 300 C and 500 C a recovery of dislocations might occur by creation of a dislocation substructure. Therefore, afterwards the reorientation of martensite becomes more easier whereas the growth of martensite plates is still prevented by this substructure. The good 1-way shape memory property of specimen 4 would be the result of the absence of martensite plate growth, the latter introducing unfavorable internal stresses.

However, the explanation based solely on dislocations cannot explain

1) the decrease of the E-modulus
2) the inhomogenous plastic flow of the c-w specimens
3) the superior 2-way shape memory properties of specimen 3

The decrease of E was already observed in other NiTi alloys (6) and was attributed to the appearance of the premartensitic phase, the so-called R-phase. The R-phase is a second order transition, which distortion growth with decreasing temperature; also it is associated with the softening of one or several phonon modes (7). This softening is the cause of the decrease of the Young's modulus. From our measurements, this would mean that more R-phase appears on c-w specimens before the martensic transformation than on annealed specimens. It is known that the presence of dislocations favours the nucleation of precipitates in lowering the nucleation energy. It could be the same for the R-phase.

It seems clear (8) that the R-phase is not a necessary step between the CS-Cl structure and the martensitic phase. One observes here also that different amount of R-phase does not change drastically the martensitic reaction.

However, it could be assumed that the R-phase is a superstructure of yet still unknown structure (9), which could be present both in martensite and in parent crystals; this phase could play an essential role in some phenomena, associated with the martensic transormation, like the nucleation of the martensite (10), the 2-ways effect (11) and the pseudo-elasticity below M_s. For the former, it is suggested that the R-phase could nucleate preferentially on dislocations and then the R-phase itself could act as nucleus for the martensite. It can be noted that this explanation is not very far from the one proposed by Clapp (12). In following this idea, the presence of the R-phase could be the key of the 2-ways effect and of the pseudo-elasticity observed below M_s. Indeed in assessing that for each variant of martensite, it exists preferentially oriented R-phase and that a martensite variant with the "right" R-phase is more stable that a variant with a "wrong" R-phase, the 2-ways effect could be due to the presence of a textured R-phase, due itself to a anisotrope network of dislocations. Also, after cold-working, the R-phase could be pinned by dislocations and then the reorientated martensite variant would be unstable. At last, the presence of the R-phase might increase the intrinsic mobility of dislocations in diminishing the Peierl's hills height by lowering the crystal symmetry of the structure and this effect could be a factor explaining the inhomogenous plastic flow.

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	Nominal composition (WT %)	M_S	c-w	Anneal
NiTi	45.5 Ti 54.5 Ni	∿ 50 C	35 %	-
NiTiCu 1	45.5 Ti 54.5 Ni 5 Cu	∿ 30 C	45 %	-
2			45 %	300 C
3		50 C	45 %	500 C
4		50 C	-	950 C
Wire a			80 %	-
b			45 %	-

TABLE: Composition, M_S temperature and thermo-mechanical treatment