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EFFECTS OF PLASTIC DEFORMATION AND TEMPERATURE ON MICROSTRUCTURAL AND MECHANICAL PROPERTIES OF A CuAlNi SHAPE MEMORY ALLOY

Carmela GURAU, Gheorghe GURAU
"Dunărea de Jos" University of Galaţi
carmela.gurau@ugal.ro

ABSTARCT

In Cu 12.88% wt.Al4 %wt. Ni shape memory alloy the influence of plastic deformation and thermal treatments on the microstructures and hardness were studied by optical microscopy, scanning electron microscopy (SEM), Vickers Hardness. The plastic deformation on the austenite (β) was studied at temperatures T>Md when transformation can occur with no stress or strain induced condition. Lattice defects were introduced into stable austenite of a Cu Al Ni alloy by a hot rolling, in one pass, and subsequent quenching after leaving the rolling cylinders. From microstructural observations it has been seen that two β_1' (18R) and γ_1' (2H) martensite phases coexists at different fractions in the undeformed and deformed states. Plastic deformation causes change in the relative amounts of β_1' and γ_1' martensites with a new morphology. The finest martensite structure has been obtained by thermo-mechanical treatment. The hardness test highlights the influence of treatments tightly correlated with the structures obtained.

KEYWORDS: shape memory alloy (SMA), martensite transformation, Cu Al Ni alloy, thermo mechanical treatment

1. Introduction

The reason for studying the thermo-mechanical treatments of Cu Al Ni shape memory alloys comes from engineering demanding for low cost, higher working temperatures than brass and *nitinol* in the same time good classical properties (i.e. hardness) and shape memory.

Thermo-mechanical treatments represent a set of operations of plastic deformation, heating and cooling – made in a certain sequence – and as a result of the final structure of the metallic material, they take place in a high density of lattice defects, associated with plastic deformation.

In the case of SMA thermo-mechanical treatment involves plastic deformation of austenite (β) at a temperature above the temperature where stress induced martensite can not appear.

Through this plastic deformation at heating is introduced certain defects which will cause the change of the martensitic transformation parameters and the martensitic structure [1-2].

Also the researches are following the increases of the mechanical properties of the alloys.

The establishment of the thermo-mechanical treatment temperatures depends on the initial phase (β): ordered, unordered, which undergoes massive transformation or which precipitates in a solid phase. Copper based alloys have an unordered structure.

Beside the importance of the M_s and M_f points at cooling and A_s and A_f points at heating, of high importance is also the M_d point placement. This point is situated above the martensitic transformation interval up to where can be obtained no stress or stress induced martensite [1]. Above this temperature the martensitic state can not be induced. It is obvious that an important role in the reversible martensitic transformation development is played by the quantity of lattice defects mainly the density of the preexistent dislocations in the austenite phase (β) and which have to interact with the moving interfaces during the martensitic transformation. A large quantity of defects introduced in the high temperature phase can favor germination of the grains or it can make sliding more difficult. Thus the A→M or M→A transformation occurs easily and through this the memories properties are improved. Also the hardness and plasticity properties will be affected.

2. Experimental research

The research program has used extruded wires 4 mm in diameter Cu Al Ni shape memory alloy prepared by classical methods at *Dunarea de Jos University of Galati.* The extruded samples of 4 mm diameter have been heated in a vertical furnace type Nabertherm for 30 min. at 850^{0}C. The samples have been introduced into the furnace at the solubilization temperature. After heating they have been rapidly immersed into ice water. Part of the extruded samples has been hot rolled on a double reversible rolling mill.

Those samples have been hot rolled in three successive passes up to 2.6 mm thickness. Some of them have been annealed to 850 C^0 and quenched in ice water. The specimens of 2.6mm thickness had been thermo-mechanically treated at high temperature respective by 1000 C^0, 950 C^0 and 850 C^0 with 20% deformation degree and cooled in ice water ride after being taken out from the rolling mill. The specimen plastically deformed at 950 C^0 have been deformed with 10%, 20% and 30% deformation degree and cooled in the same conditions.

The plastic deformations were done on a duo reversible rolling mill, home made, in one pass. The heating furnace was placed in front of the rolling mill.

The sample route has been protected by the ceramic tube. The loss of heat through radiation is limited, aspect that posed more problems as the rolled sample thickness decreased. The rolling cylinders were preheated with methane gas burner.

The high thermo-mechanical treatment domain limits for the Cu Al Ni alloy were fixed according to the melting temperature and the minimum temperature was chosen to have no cracks in the sample.

$850^0C \leq T < T_{melting}$

After rolling, the subsequent quenching is essential. It can be considered that the alloy leaves the rolling mill at about the same temperature at which it entered do to adiabatic heating.[1]

The samples were analyzed by optical and electronic microscopy and hardness test. Prior to investigate the specimen was metallographically prepared.

2.1. Analysis of the crystalline structure by optical microscopy

The microstructural study has been carried out with an optical microscope Olympus type equipped with a digital camera and connected to a computer at *Dunarea de Jos University of Galati*. The sample surfaces have been metallographically prepared and attacked by Klemm's III.

2.2. Analysis of the crystalline structure by scanning electron microscopy (SEM)

The microstructural study has been carried out with an optical microscope Philips type at *Dunarea de Jos University of Galati*. Also the sample surfaces have been metallographically prepared and attacked by $FeCl_3$ in water solution.

2.3. Hardness test

The hardness tests have been performed with a hardness meter Vickers at *Dunarea de Jos* University of Galati. The sample surfaces have been metalographically prepared.

3. Results and discussions

The Cu Al Ni SMA alloy that undergoes this type of thermo-mechanical treatment has a critical point for transformation, M_f above the room temperature. The introduction of a volume of defects in the initial phase has been done by hot rolling. The applied thermo-mechanical treatment consists in heating in the stable domain of the initial phase then plastic deformation at high temperature followed by quenching.

The procedure allows the variation of temperature also the application of range of deformation degrees. There are 3 intervals:

a) *High temperature interval up to the temperature where the stress induced martensite can appear ($T > M_d$).*

In this temperature interval, the austenite is thermodynamically stable. Above the critical temperature A_f, inside the alloy the stable solid solution is type B_2 unordered. In the austenitic transformation domain takes place the arrangement of this solid solution into a type DO3 long ordered solution. Under the critical temperature A_s inside the ordered solid solution, the martensite b_1' incipient crystal germs appear. Also it can appear metastable compounds.

b) *The interval between the temperature up to which stress induced martensite appears and the finish martensitic temperature ($M_d \leq T \geq M_f$).*

In this temperature interval the thermodynamic stable austenite has been converted to martensite. Through application of the plastic deformation on the austenitic phase alloy, the initial transformation into stress induced martensite has been slowed. Also we will have transformations and reorientations in the partially transformed martensite:

$\beta + M^{+/-} \rightarrow (\beta \rightarrow M^{+}) + (M^{+/-} \rightarrow M) \rightarrow M$ [1]

c) *Low temperature interval under the finish martensite critical point ($T > Mf$).*

In this temperature interval the martensite is thermodynamically stable. This interval corresponds to the last part of the thermo-mechanical treatment, rapid quenching occurs. There are only reorientations of the martensite: $M^{+/-} \rightarrow M^{+} \rightarrow M$ [4].

In order the study has been focused in the first temperature domain because most of reports refers only to the third domain.

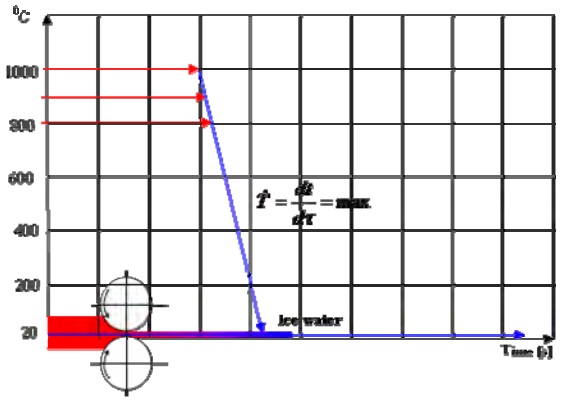


Fig. 1. *The thermo-mechanical treatment scheme*

Thus the increase in the deformation degree and the decrease deformation temperature favors the random distribution of the dislocation in the material volume.

We assume that all the induced dislocation in the initial phase will be found in the martensite which will be strengthen. During the reversible transformation $b \leftrightarrow M$ the Burgers vectors must undergo the $b(b) \leftrightarrow b(M)$ transformation. In both phases the dislocations must be connected to the limits of antiphase domains. That is necessary for a reversible and thermoelastic process, mandatory for the shape memory alloys [1].

The austenite dislocations favor martensite at cooling moving the Ms point towards higher values.

$$Ms = Ms_0 + M_{sr}$$

3.1. Optical microscopy

The specimens rolled at 1000^{0}C, 950^{0}C, 850 ^{0}C were subjected to metallographic study, in order to investigate the influence of thermal treatment on the grain size responsible for mechanical properties and memory properties of the alloy.

From all microstructural observations, it is seen that b_1'*(18R)* and g_1' *(2H)* martensite phases coexist at different fractions also in deformed states. In the figure 2a, these is preponderantly a typical martensite morphology b_1'*(monoclinic),* zig-zag structures [3]. The originate grains have a polygonal shape, linear smooth faces and coarse needles. In the figure 2b and 2c predominantly are variants of the g_1' *(orthorhombic)* martensite, along with smaller amounts of b_1' martensite.

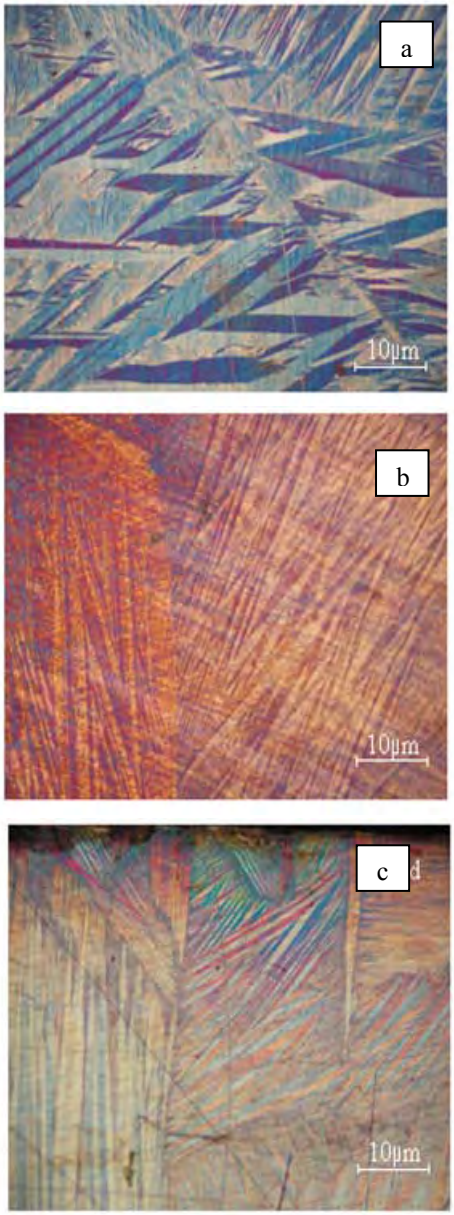


Fig.2. *Microstructures of hot rolled alloy at 20% deformation degree*
a- 1000^{0}C,b- 950^{0}C, c- 850^{0}C

These results show that the decrease of temperature induces changes in the relative amounts of the b_1' *and* g_1' martensites. There are structures with more fine needles. The g_1' *(2H)* martensites have been germinated on b_1'. Plastically deformed specimens at 850^{0}C have curved martensite perpendicular to rolling direction. The grains boundaries become rugged. In each analyzed sample the grains have generally large sizes. The average

grain sizes do not present large differences among the samples thermo-mechanically treated at the same strain but in three different plastic deformation temperatures.

The influence of strain is seen in figure 3. It can be observed that structures are obviously inhomogeneous.

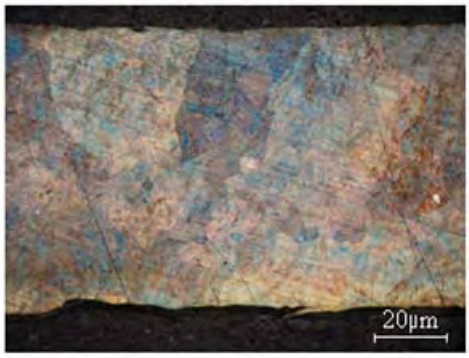
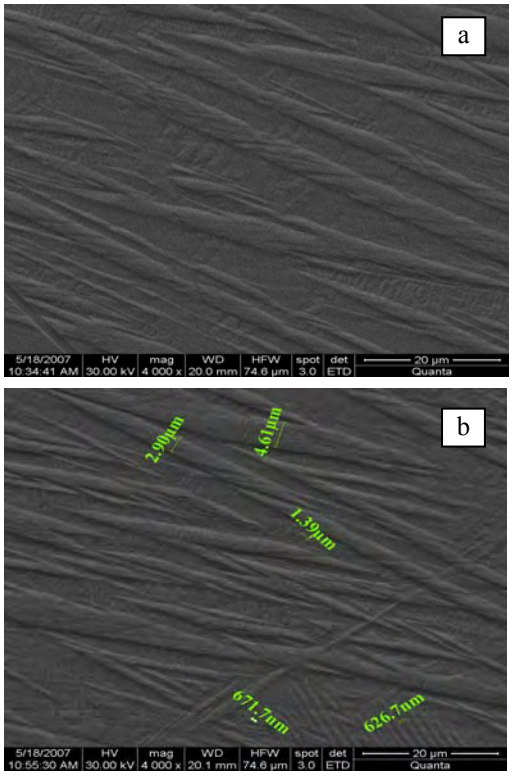


Fig3. *Microstructures of hot rolled alloy at 30% deformation degree and* 950^{0}C

There are large grains beside small grains elongated in the rolling direction. The average of grain size presents significant difference at different deformation degree. The high refinement can be observed at 30% deformed.

3.2 Scanning electron microscopy

In figure 4are presented SEM observations for the samples rolled at different temperatures but at the same deformation degree.



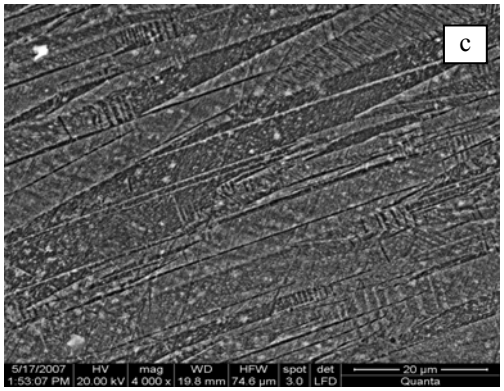


Fig.4. *SEM microstructures of hot rolled alloy, at 20% deformation degree and*
a- 1000^{0}C,b- 950^{0}C, c-850^{0}C

The pictures highlight two typical martensites morphologies that coexist in the relative amounts alloy for different plastic deformation temperatures. In conditions of decreasing temperatures it can be observed a refinement of needles. In figure 4b the needles for the specimen deformed at 950^{0}C have sizes ranging from 4.61 μm to 626.7nm. It can be also observed that those specimens contain mechanical twins especially the sample from picture 4c thermo-mechanically treated at 850^{0}C and 20% deformation degree [4].

3.3. Hardness test

Figure 5 gives the hardness test results for the sample Cu Al Ni thermo mechanically processed at different temperatures (1000^{0}C, 950^{0}C, 850^{0}C), 20% deformation degree.

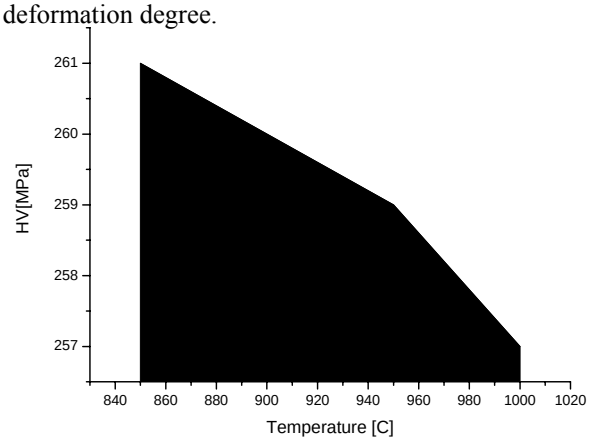


Fig.5. *Hardness versus deformation temperature*

Figure 6 gives the hardness test results for the sample Cu Al Ni thermo mechanically processed at

950^0C, in range of 10%, 20%, 30% deformation degree.

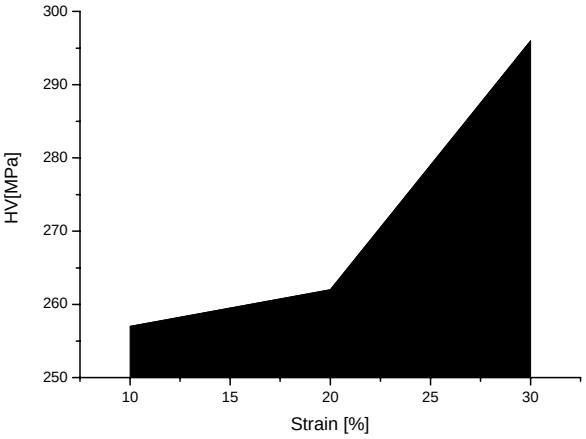


Fig.6. *Hardness versus strain*

It can be observed that the influence of strain is more important in increasing hardness values then the influence of plastic deformation temperatures.

The lowest hardness was obtained for the material subjected to quench after extrusion (250MPa). The specimen with higher hardness was obtained by thermo mechanical treatment from 950^0C with 30 deformation degree (296 MPa). Those values are tightly correlated with obtained structures.

4. Conclusions

1. The thermo-mechanical treatment applied in initial phase of shape memory alloy (b) can change the general properties of the alloy having a positive impact on the shape memory applications.

2. High dislocation density in the initial phase improves the martensitic transformation.

3. The martensitic transformation points are established through the introduction of preferential dislocation distributions for certain martensitic variants.

4. The optical microscopy highlights the coexistence within the alloy of two types of martensite b_1'*(18R)* and g_1' *(2H)*. The heat treatments applied affect the structure and properties of the alloy and implicitly its memory features. The finest martensitic structure has been obtained by thermo-mechanical treatment (T=950^0C, ε=30%);

5. There was a grains refinement accompanied by the elongation phenomenon of the initial austenitic grains. The g_1' martensites have been germinated on b_1' martensites as a results of increasing deformation degree and decreasing plastic deformation temperature.

6. The hardness test highlights the influence of the thermal treatments tightly correlated with the structure obtained.

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