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# Variability in mechanical properties and microstructure characterization of CuAlBe shape memory alloys for vibration mitigation

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**Abstract.** Shape memory alloys (SMA) have been emphasized, studied and understood in the controlled world of the laboratory. Any attempt to implement one of these alloys in engineered products requires a jump from the controlled world of the laboratory to the actual environment of the application. The first step is to move from single grain specimens to multigrain samples. One works with a material for which any stock is different from that previously available. This paper reviews the milestones in the familiarization process the authors had to overcome during their cooperation within a project funded by the European Union. The main items cover transformation temperatures, thermal treatment and properties understanding.

**Keywords:** shape memory alloys testing specimens; thermal treatment; transformation temperatures; vibration mitigation.

## 1. Introduction

Within the EU FP6 project WIND-CHIME the authors worked with alloys, in the austenitic state, of both Nickel-Titanium and of Copper, Aluminium and Beryllum (Auricchio, *et al*. 2001, Andrawes and DesRoches 2007). The acquisition aspects related with the first alloy are treated in different papers (van der Eijk, *et al*. 2006 and 2007). The Copper based alloy was studied in detail due to its lower cost and mainly because it is preserving its austenitic state in a broader window of temperatures (Casciati and Faravelli 2004, Casciati and Faravelli 2003, 2006 and 2007, Casciati, *et al*. 2007). Its drawback, being the alloy based on Copper, is sensitive to humidity, when present.

The owner of a construction company, years ago, asked the first author where it was possible to buy some kilos of shape memory alloy for building a prototype. If successful, he would have acquired a large amount of alloy from an international producer. The first author had seen grams of it being produced in a research laboratory and promoted a meeting between the company manager and the laboratory head. An order was placed and, with a delay judged excessive by the construction company, the alloy was produced and delivered. It was behaving in all ways except in the desired one. It happened

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that moving from the production of small amounts to one of large ingots, the laboratory was unable to control some precipitates and this prevented the alloy from having its desired (expected) behaviour. The tale suggests that the production of shape memory alloy must be demanded to professionals: the ingredients are well known, but to mix them is not straightforward.

Based on this experience, the authors relied on a well identified French producer (the names are ignored to preserve a sort of "being free from commercialism", but the interested reader can ask directly the authors) which already produced for the team a stock of several kilos of alloy within the previous FP5 EU funded project CHIME. Part of this material (stock 1) was available at the laboratory of the first author, but the developments of WIND-CHIME demanded further quantities of material and different shapes (bars of different diameter and so on). When the order was ready to be placed, it turned out that the French producer had dismounted the production unit, sold the furnace to a German company and passed the shape memory alloy production to a different French company (with different tools and people). This second producer sent a first sample of material in the form of bars of diameter 15 mm (stock 2). Then a larger order was placed aiming at the production of several kilos in ingots to be worked later (stock 3). In the meantime the authors had the chance to obtain samples of two further kinds of alloy, the first coming from a third producer and the other from the same new French company. Formally, the weight composition of the material was always the same. This paper discusses their quite different properties.

## 2. The alloy

The alloy has label AH140 and it is made by Copper, Aluminium and Beryllium, (11.8 wt% Al, 0.5 wt% Be). Before use, the alloy requires a preliminary thermal treatment ("aging") and a preliminary mechanical treatment ("training").

The thermal treatment consists of three steps: i) the alloy item is put in the oven at 850 $^{o}$C for 10 minutes (or better 30 minutes per each inch (2.54 cm) of diameter of the specimen), ii) air cooled to room temperature and then iii) maintained at 100 $^{o}$C for a period that should theoretically be of two months. This period is often reduced to minutes (say 120 minutes), with the consequences that will be discussed later. The literature shows a sensitivity of the process to the actual value of the oven temperature, which must be carefully checked.

The mechanical treatment was set mainly for wires and bars: They undergo cycles of loading-unloading, from zero up to a fixed value of the strain: 10 very slow cycles could be sufficient, while faster cycles should count up to 50 in order to complete the treatment.

## 3. The laboratory set-up

This paper reports the laboratory results coming from four different instruments: 1) differential scanning calorimeter (DSC); 2) optic microscopy; 3) SEM and EPMA analysis and 4) universal testing machine.

### *3.1. Differential scanning calorimeter*

The differential scanning calorimeter test (DSC) estimates the temperature of phase transition. A suitable sample, of weight, say, 20 mg, is prepared, treated by chemical etching (to remove superficial

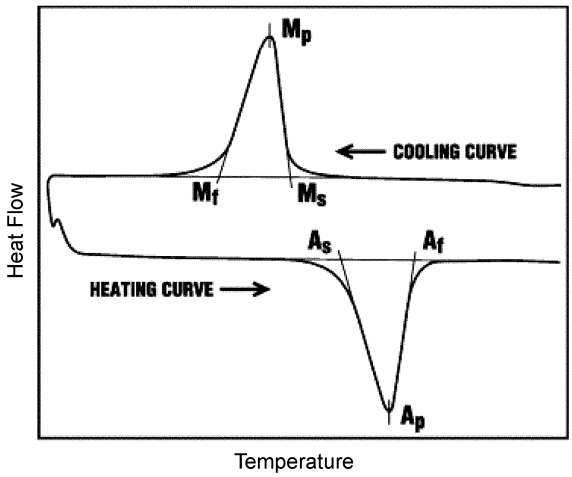


Fig. 1 Schematic DSC curve illustrating the method with which the phase transformation temperatures are measured

martensite) and subjected to a variation of temperature.

Differential scanning calorimetry is a technique used to determine the variation of thermal flows emitted or received by a sample when subjected to a temperature program in a controlled atmosphere. When heating or cooling, any change occurring in the material is accompanied by an exchange of heat: DSC enables the temperature of this transformation and the quantity of the heat produced to be determined. The DSC equipment at SINTEF is a Seiko DSC220C with exstar 6000 Thermal Analysis system software.

The results of the test comes in the form of diagrams: the temperature in the abscissa and the heat in the ordinate. Recall that austenite is stable for low stress and high temperature, while the martensite is stable for high stress and low temperature. The transformation from austenite to martensite and the reverse transformation, from martensite to austenite, do not occur at the same temperature. The complete thermal cycle is characterised by four temperatures: martensite finish temperature $M_f$, martensite start temperature $M_s$, austenite start temperature $A_s$ and austenite finish temperature $A_f$. In both cases, a third value, characterised by the index $p$, can be added: it represents the temperature at which the heat flow reaches its peak.

Therefore, the phase transformation temperatures are conveniently represented by the graph in Fig. 1.

### *3.2. Optical microscopy, SEM and EPMA analyses*

The degree of heterogeneity of the material (due to the production technology) and the shape and dimension of the grains can be assessed by microscopy analyses. The specimens were mounted, grinded and polished. The microstructure was investigated without etching in polarised light.

In addition, the laboratory of the second author gives access to a scanning electron microscope (SEM, model Hitachi, S-4300SE) and to an electron probe micro-analyzer (EPMA) An electron probe micro-analyzer or microprobe is basically a scanning electron microscope designed and optimized for X-ray analysis of elements from very small areas. The JXA-8900 instrument is equipped with 4 wavelength dispersive X-ray spectrometers (WDS) and an energy dispersive X-ray spectrometer (EDS). This combination can simultaneously analyse up to 12 elements plus collect image signals from backscatter and secondary electron detectors.

### 3.3. Universal testing machine

All the mechanical tests were carried out on the bi-axial universal testing machine MTS 858, Minibionix II: hydraulic linear and torsion actuators are driven in span/angle, strain or load/moment control by a suitable MTS software. A thermal chamber allows one to carry out tests at different temperatures.

The strain can be assessed by the extensometer 632.31F-24 which works on a basis of length 10 mm.

## 4. Stock 1 – the initial production

Stock 1 denotes the material which was available at the laboratory of the first author when the project started. As said, the material was acquired from a first French producer which later sold this specific production.

Three samples were prepared from material in stock 1 for the DSC and micro-analyser analyses. They come all from the wire of diameter 1 mm, in particular:

a) from the alloy as delivered by the producer;
b) from the alloy heat treated: at 850 ºC in furnace for 10 minutes, water quenched and aged at 100 ºC for 120 minutes;
c) from an alloy wire first aged and then trained by cycles of loading and unloading.

### 4.1. Optical microscopy

The specimens were mounted, grinded and polished. The microstructure was investigated without etching in polarised light. The micrographs are shown in Fig. 2. The as-produced specimen is fully austenitic and the grains are stretched due to the production process. The heat treatment and aging lead to re-crystallisation and grain growth. After training, more grain growth is observed and some martensitic needles can be detected.

### 4.2. Scanning electron microscopy

The samples were investigated in a Scanning Electron Microscope to search for contaminating particles like oxides and sulphides which could degrade the mechanical properties. No such second phase particles were found.

### 4.3. Differential scanning calorimeter (DSC)

The results are arranged in Table 1. The martensite start and finish temperatures of the heat treated and trained material are close to the temperatures specified by the producer. The austenite start and finish temperatures are slightly lower than specified. Considering that the austenite finish temperature is well below room temperature, the martensitic needles observed in the microstructure of the trained material, shown in Fig. 2c), are all due to the training stresses.

### 4.4. Mechanical testing

The alloy of stock 1 was produced in a large amount and the ingots were worked to obtain several

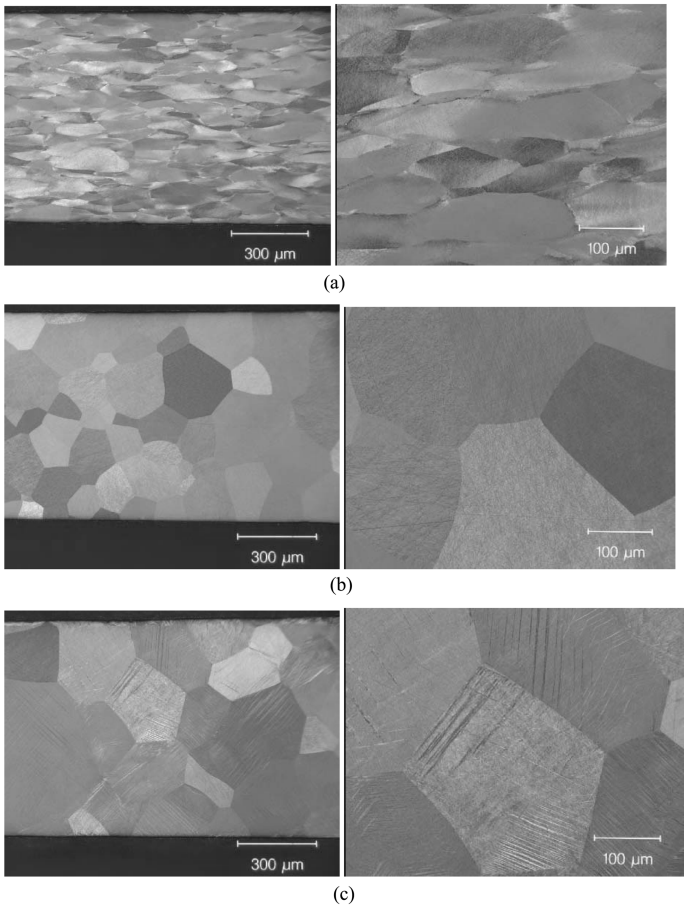


Fig. 2 Optical micrograph of the material (a) in the as-produced condition; (b) in the heat treated and aged condition; (c) in the heat treated + trained condition

Table 1 Measured phase transformation temperatures for the three samples of stock 1

| Sample | $M_s$ | $M_f$ | $A_s$ | $A_f$ |
|---|---|---|---|---|
| As-produced | -46 | -55 | -25 | -18 |
| Heat treated and aged | -27 | -34 | -23 | -13 |
| Heat treated, aged and trained | -23 | -47 | -30 | -9 |
| Specified by the producer | -18 | -47 | -20 | +2 |

shapes as wires (of diameters 1 and 2.85 mm), bars (of diameters 5.3 mm and 15 mm) and plates. Tests were carried out on specimens from all the element shapes. Fig. 3 just provides a test with several loading-unloading cycles for the bar of diameter 5.3 mm in the two cases: untreated (a) and treated (b). The material is treated in the standard way. The strain is measured by the extensometer. The tests in Fig. 3 are driven in strain control (speed 0.005 [1/s]) during the loading and in load control during the unloading. The unloading time is set equal to the loading time for each cycle.

The following aspects are worth noticing:

1) the plateau is not actually flat (horizontal);

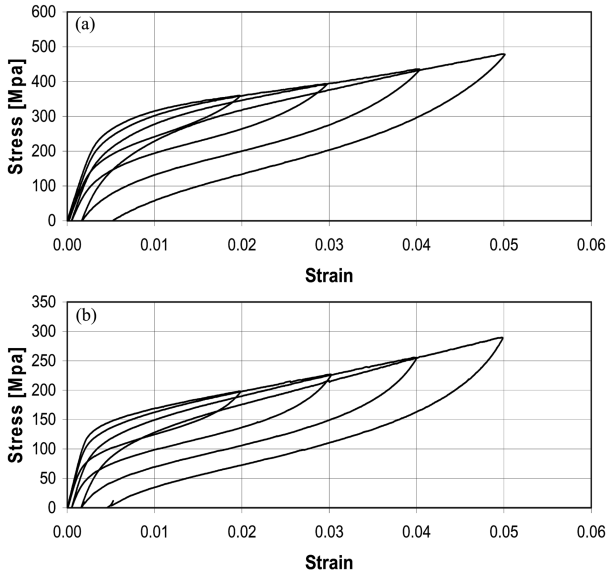


Fig. 3 Tension test for specimens (bars of diameter 5.3 mm) of stock 1: (a) untreated case and (b) treated case

2) the knee between the initial linear branch and the plateau occurs at 250 MPa in the untreated case, but at 120 MPa for the treated specimen;
3) as repeated cycles of increased strain progress, an increasing residual strain is detected, due to martensite which is not recovered. For the cycles of 5% maximum strain one sees a residual displacement of 0.5%.

## 5. New production vs. old production

After the three CuAlBe specimens from the first French producer investigated in the previous section, eight CuAlBe specimens are investigated in this section. The specimens had the following designations:

The meaning of the labels A and B will be explained later in this section. By contrast, label 1 refers to a specimen taken from a bar of diameter 15 mm belonging to stock 1 from the first producer. A second acquisition of bars of diameter 15 mm (stock 2) occurred from the second producer and was followed by a third one (stock 3), to which label 3 corresponds. Unfortunately all the material belonging to stock 2 was tested before realizing the need of the investigations reported in this paper and no micro-structure characterization is available for it.

### *5.1. Optical microscopy*

The specimens were mounted, grinded and polished. The microstructure was investigated without etching in polarised light. The micrographs are shown in Figs. 4 to 11. There are large differences in the microstructure of the untreated specimens. The AU specimen appears to contain martensite. Grain growth occurs following the thermal treatment in all cases.

Note that a large grain size is known to have a positive influence on the damping properties of CuAlMn alloys (Sutou, *et al*. 2006).

Table 2 Designation of the specimens analysed in section 5

| SAMPLE | Abbreviation |
|---|---|
| 1 untreated | 1U |
| 1 treated | 1T |
| 3 untreated | 3U |
| 3 treated | 3T |
| A untreated | AU |
| A treated | AT |
| B untreated | BU |
| B treated | BT |

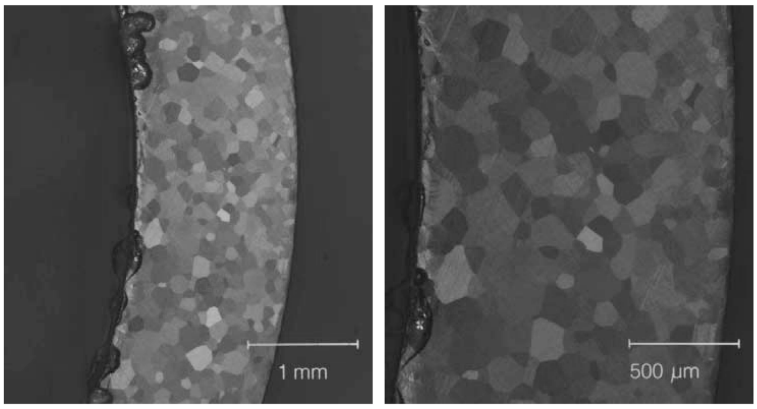


Fig. 4 Optical micrograph of the 1U specimen

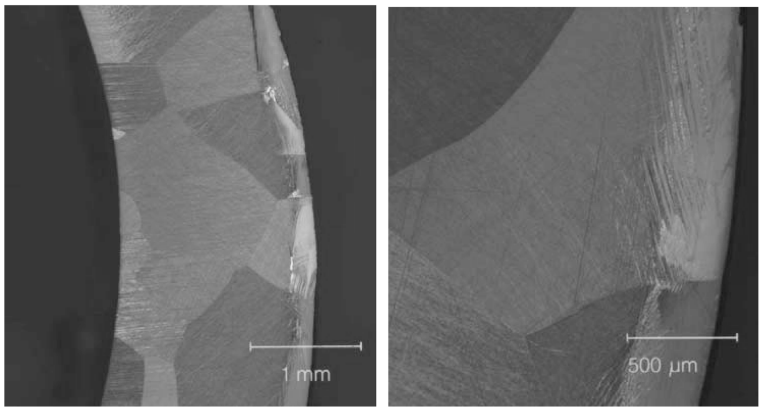


Fig. 5 Optical micrograph of the 1T specimen

### *5.2. Scanning electron microscopy/microprobe analyses*

The samples were investigated in a Scanning Electron Microscope/Microprobe with four Wavelength Dispersive X-ray Spectrometers (WDS). Quantitative analyses were taken from the materials from

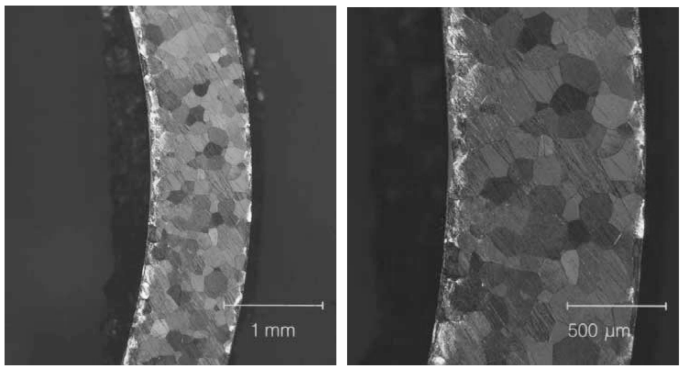


Fig. 6 Optical micrograph of the 3U specimen

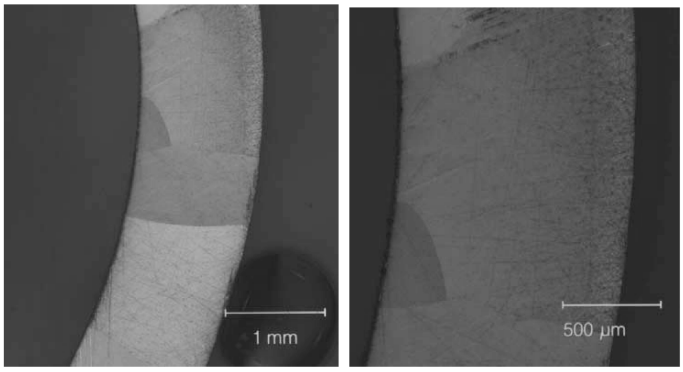


Fig. 7 Optical micrograph of the 3T specimen

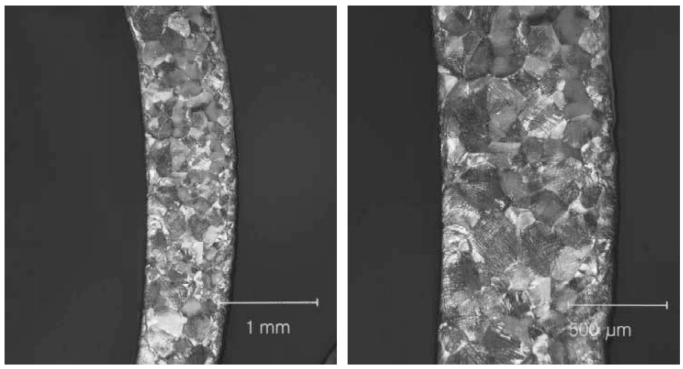


Fig. 8 Optical micrograph of the AU specimen

both producers. Beryllium could not be quantified in a reliable way because it is a light element. The current samples (1, 3, A and B) had all identical chemical compositions. There is a slight, but statistically not significant, difference in the chemistry between the materials from the two producers as in Table 3. According to the specifications the materials should contain 11.8 wt% Al and 0.5 wt% Be.

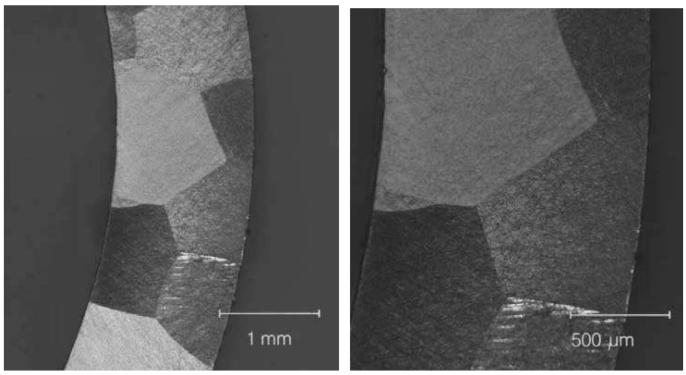


Fig. 9 Optical micrograph of the AT specimen

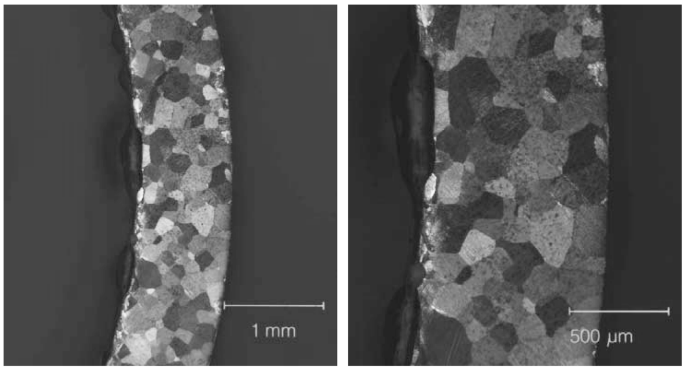


Fig. 10 Optical micrograph of the BU specimen

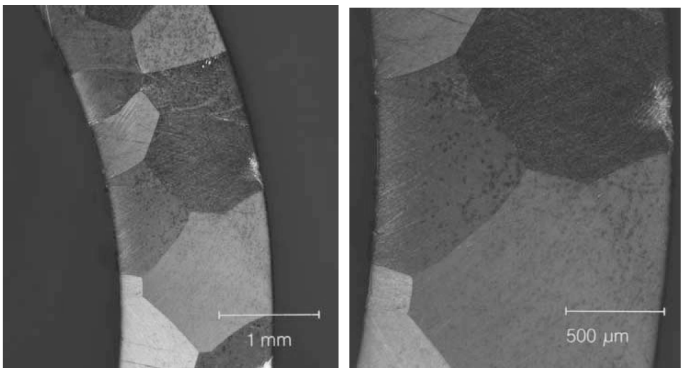


Fig. 11 Optical micrograph of the BT specimen

Table 3 WDS analyses from the two producers

| Wt% | Al | Cu |
|---|---|---|
| Specimens in section 4. | 12.7±0.5 | 87.3±0.4 |
| Specimens in section 5. | 13.0±0.1 | 87.0±0.2 |

Table 4 Measured phase transformation temperatures for the two producers samples

| ºC | $M_s$ | $M_p$ | $M_f$ | $A_s$ | $A_p$ | $A_f$ |
|---|---|---|---|---|---|---|
| Values specified by the producer | -18 | | -47 | -20 | | +2 |
| 1U | -27 | -31 | -42 | -20 | -13 | -8 |
| 1T | -13 | -17 | -27 | -11 | -5 | +2 |
| 3U | -28 | -35 | -45 | -27 | -17 | -12 |
| 3T | - | -25 | -31 | -17 | -10 | -4 |
| AU | | -25 | -33 | | | |
| AT | -18 | -23 | -35 | -16 | -8 | 0 |
| BU | -38 | -57 | -73 | -41 | -22 | -15 |
| BT | -21 | -24 | -34 | -14 | -7 | -1 |
| 1st producer alloy as produced | -46 | -50 | -55 | -25 | -21 | -18 |
| 1st producer alloy heat treated and aged | -27 | -31 | -34 | -23 | -17 | -13 |
| 1st producer alloy heat treated and trained | -23 | -37 | -47 | -30 | -17 | -9 |

### 5.3. Differential scanning calorimeter (DSC)

During Differential Scanning Calorimetry (DSC), the phase transformation temperatures are measured as illustrated in Fig. 1. The results are listed in Table 4. The martensite start temperatures of the treated material are close to the temperatures specified by the producer. The austenite start and finish temperatures are slightly lower than specified. Considering that the austenite finish of the 1T material is 2 ºC, it is surprising that some martensitic needles are observed in the microstructure of the 1T material shown in Fig. 5. Indeed, differently from section 4.3, no training process was justifying these needles.

### 5.3. Mechanical testing

Tests on material labelled as 1, treated and aged, covers the same material of section 4. But the specimens come from bars of diameter 15 mm!.

Fig. 12 just provides the test to rupture (a) and a test with several loading-unloading cycles (b) for a pipe section specimen obtained by the bar of diameter 15 mm (internal diameter 12.5 mm). The material was treated shorter than usually. The strain is measured by the extensometer, the test in Fig. 3a) is conduced in span control (with speed 0.08 mm/s). The test in Fig. 3b) is driven in strain control (speed 0.001 1/s) during the loading and in load control during the unloading. The unloading time is set equal to the loading time for each cycle.

The following aspects are worth noticing:

1) the plateau is not actually flat (horizontal);

2) the knee between the initial linear branch and the plateau occurs at 60 MPa;

3) the rupture strain is 9%;

4) as repeated cycles of increased strain progress, an increasing residual strain is detected, due to martensite which is not recovered. For the cycles of 4% maximum strain one sees a residual displacement of 0.3%; for 5% it becomes 0.9%!.

Fig. 13 provides the same plots obtained when testing pipe specimens from stock 3. In this case one sees some aspects of interest (flatness of the plateau, recovering of the martensite fraction, i.e., of the residual displacement) improved, a more brittle behaviour but mainly a plateau level significantly higher, say doubled!

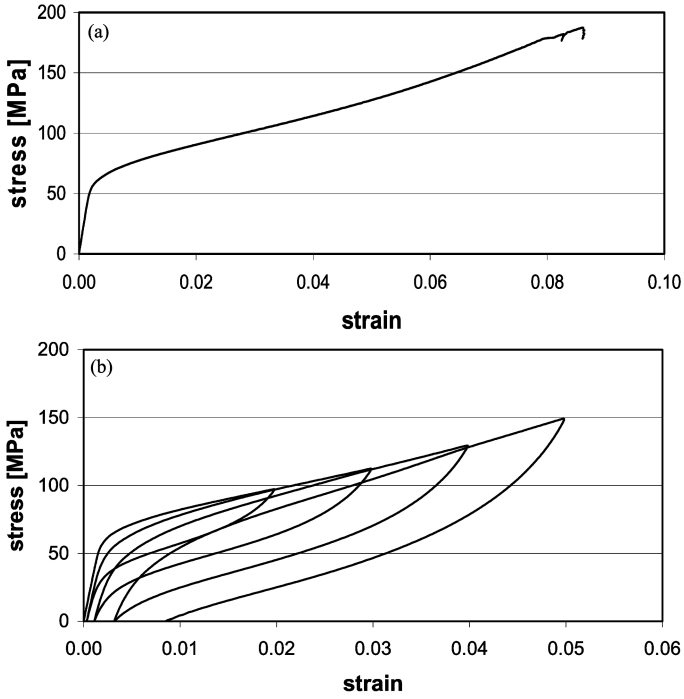


Fig. 12 Tension test for specimens of diameter 15 mm from stock 1: (a) up to rupture and (b) with loading-unloading cycles

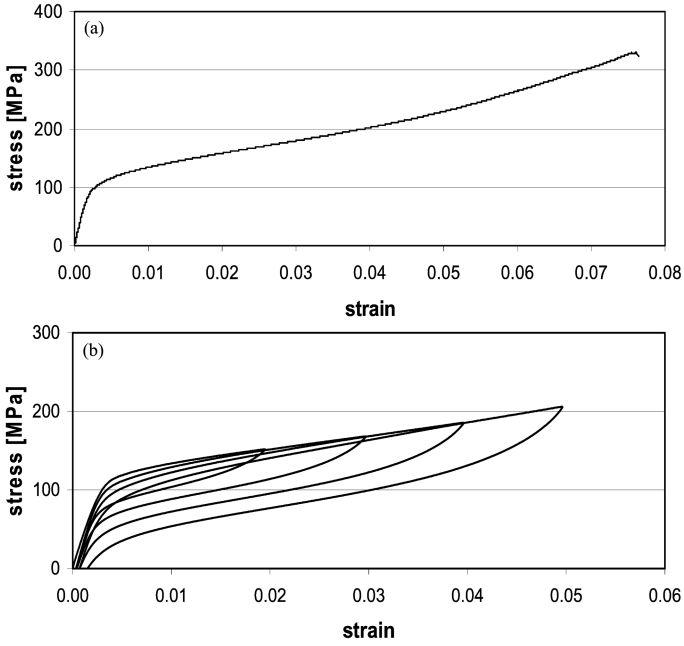


Fig. 13 Tension test for specimens of stock 3: (a) up to rupture and (b) with loading-unloading cycles

### 5.4. Discussion

The second producer was required to justify the different performance of its alloy, ordered as a further amount of the alloy in stock 1. The answer (Hautcoeur, 2006)) consists of the following items:

1) "the delivered material is strictly identical to that from" the first producer;
2) "the label AH140 denotes a casting from the first producer from which the last bars ("bilettes" in French) were acquired. Therefore, one is speaking of the same alloy";
3) "the transformation process of the bar is also the same: same heating temperature, same flow speed, same pressure. But the machines are different.";
4) "it is likely that the structure (size of the grain) be slightly different. If so, you can modify the heat treatment conditions (duration or temperature) to have the wished properties";
5) "two short bars of diameter 15 mm are delivered to you: the first comes all from the first producer; the second was processed by the new one".

These two bars provided the samples above denoted as A and B, respectively. Figs. 14 and 15 provide the results of tension tests for the untreated and treated cases. When untreated the two alloys are similar, but after the heat treatment material A shows the wished behaviour (flat plateau and full recovery of the strain after unloading) but alloy B does not. The producer was unable to justify this difference except for this: something similar to the thermal treatment (but not the aging) was already carried out by the producer of sample A.

In conclusion, the main differences of behaviour of the alloy depend on the heat treatment, which sometimes is performed directly by the producer without clear specifications. In particular the surprising difference between the results in Figs. 12 and 13 was simply due to the combination of two facts: the producer already carried a thermal treatment on the large diameters bars in Stock 1 and the thermal treatment by the author was shorter than due. A witness of this, however, is easily readable in the results of Table 4.

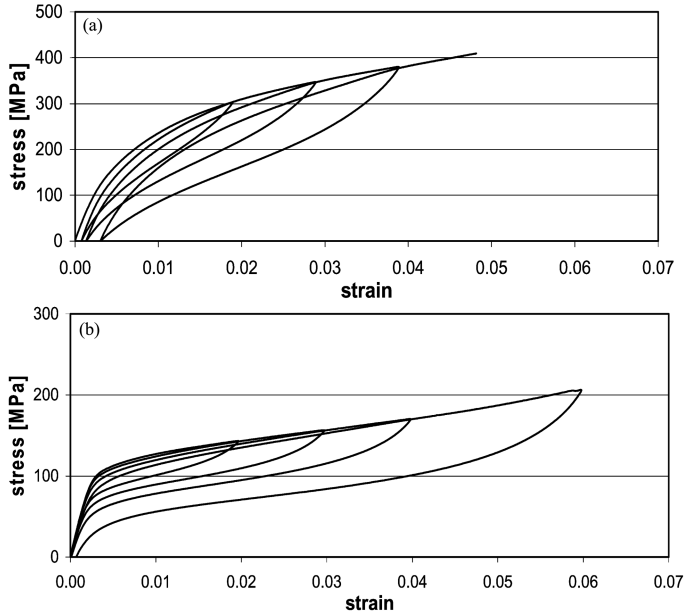


Fig. 14 Tension test for the specimen labelled as A: (a) untreated (the test was interrupted before the last unloading); (b) treated

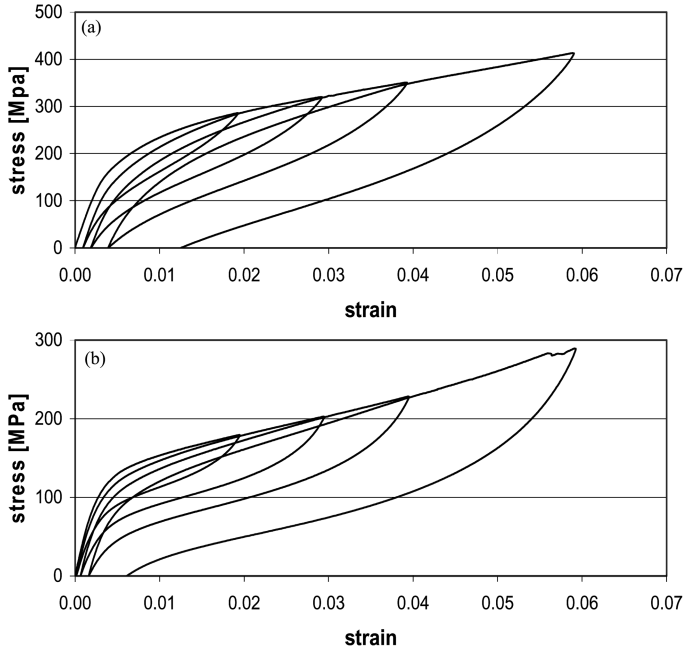


Fig. 15 Tension test for the specimen labelled as B: (a) untreated; (b) treated

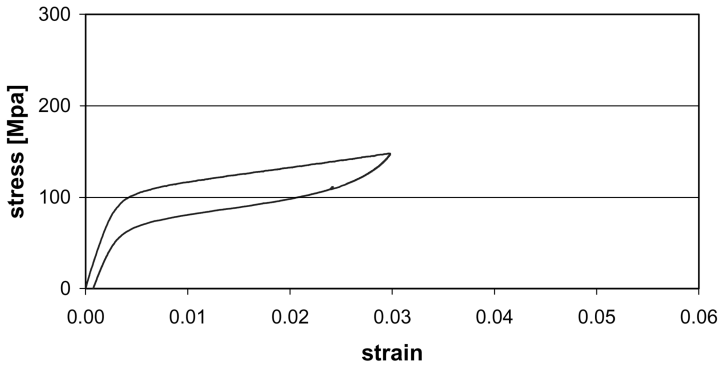


Fig. 16 Loading-unloading cycle up to 3% of strain of a specimen from stock 3 aged 1 month

Moreover, the slope of the plateau is significantly affected by the aging process, as well as the stress value at the knee. Fig. 16 shows the response to a single loading-unloading cycle of a specimen from Stock 3 aged at 100 $^{o}$C for 1 month. The graph emphasizes the lower stress value at the knee and the flatter plateau.

## 6. Further acquisitions

### *6.1. Diameter 2.5 mm*

As an exchange with a different laboratory, one meter of alloy nominally equal to the one produced by the two French producers was made available to this study (Torra, 2006). Fig. 17 gives the results of

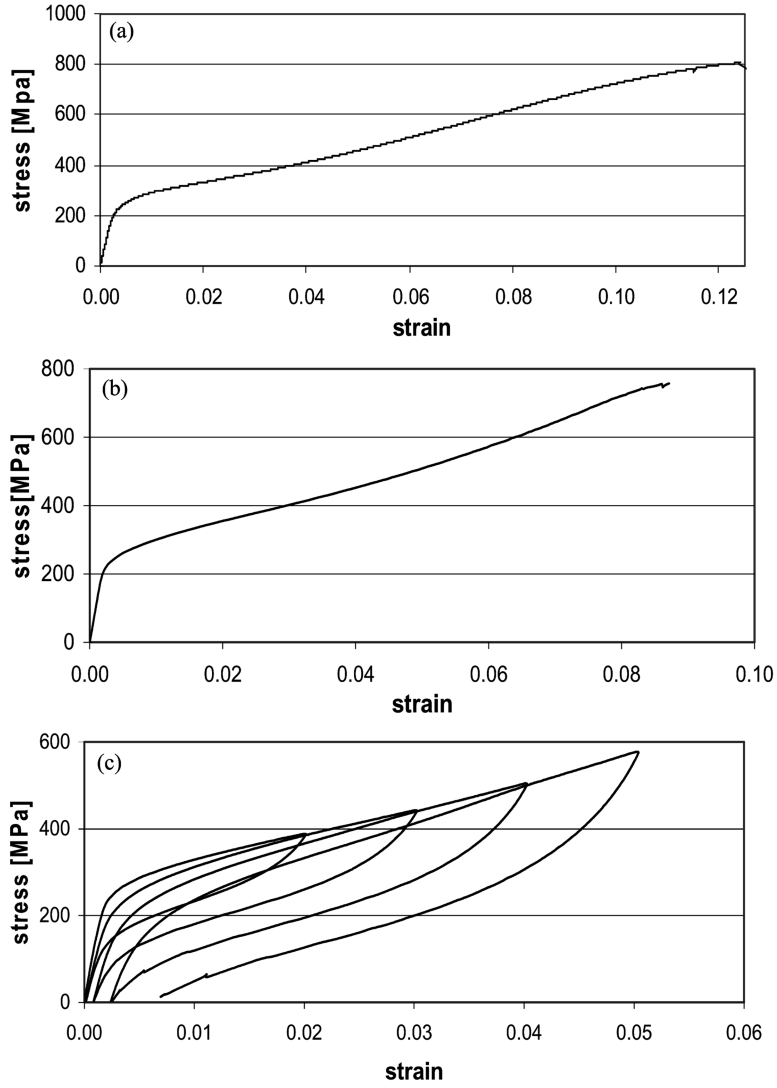


Fig. 17 Tension test for specimens of a third producer alloy: up to rupture untreated (a) and treated (b) and with loading-unloading cycles (c)

three tests carried out on the untreated (17a) and treated alloy (17b and c). It is seen that the plateau knee occurs for a value of stress much higher than the values of sections 4 and 5. Also it does not change after the thermal treatment; only the rupture strain decreases after the treatment. A 0.6% residual strain is found after a cycle up to 5% strain.

### 6.2. Diameter 3.5 mm

For the final applications within the project WIND-CHIME, it was decided to acquire from the second producer material formed in a wire/bar of diameter 3.5 mm. The untreated material was submitted to the usual microscopy and DSC test. A sample of wire of diameter 2.85 mm coming

Table 5 Transformation temperatures for the new acquisitions

| | $M_s$ | $M_p$ | $M_f$ | $A_s$ | $A_p$ | $A_f$ |
|---|---|---|---|---|---|---|
| AH140 - 3.5 mm | -20 | -29 | -38 | -19 | -12 | 0 |
| AH140 - 2.85 mm | -28 | -35 | -40 | -20 | -16 | -11 |
| AH140 - new | -128 | -129 | -130 | -14 | -7 | -1 |

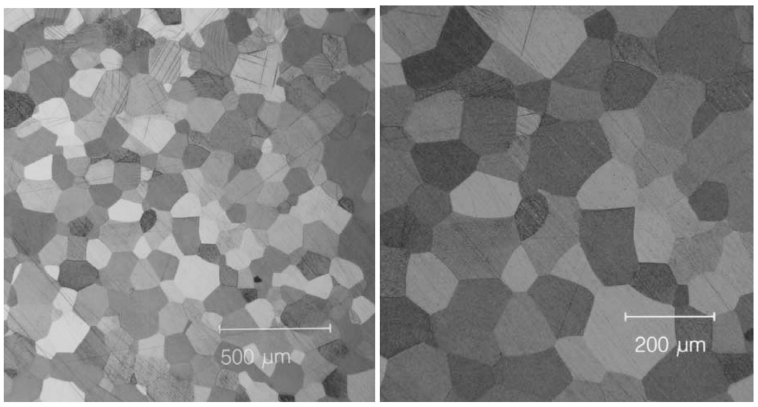


Fig. 18 Optical micrograph AH140 3.5 mm

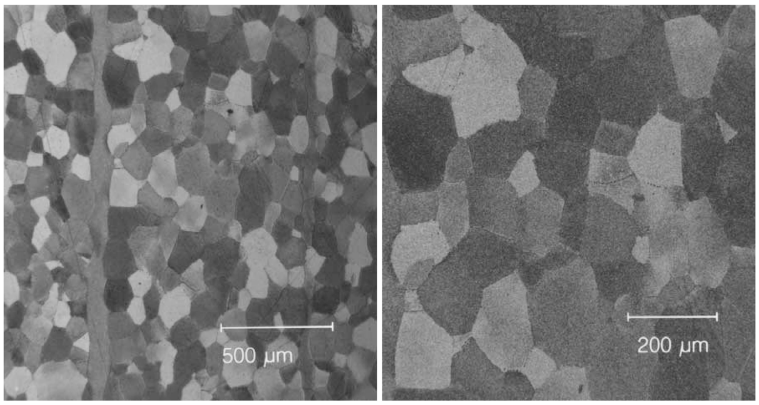


Fig. 19 Optical micrograph AH140 2.85 mm

from the old Stock 1 was also submitted to the same tests for comparison. The results are collected in Table 5.

Figs. 18 and 19 compare the two materials. Fig. 20 contains the result of the mechanical tests on the new stock alloy.

### *6.3. Material at very low martensite transformation temperature (M)*

Once again as a result of exchanges between laboratories, a further alloy, labelled M, coming in bars of diameter 3.5 mm was made available (Torra 2007). The tranformation temperatures

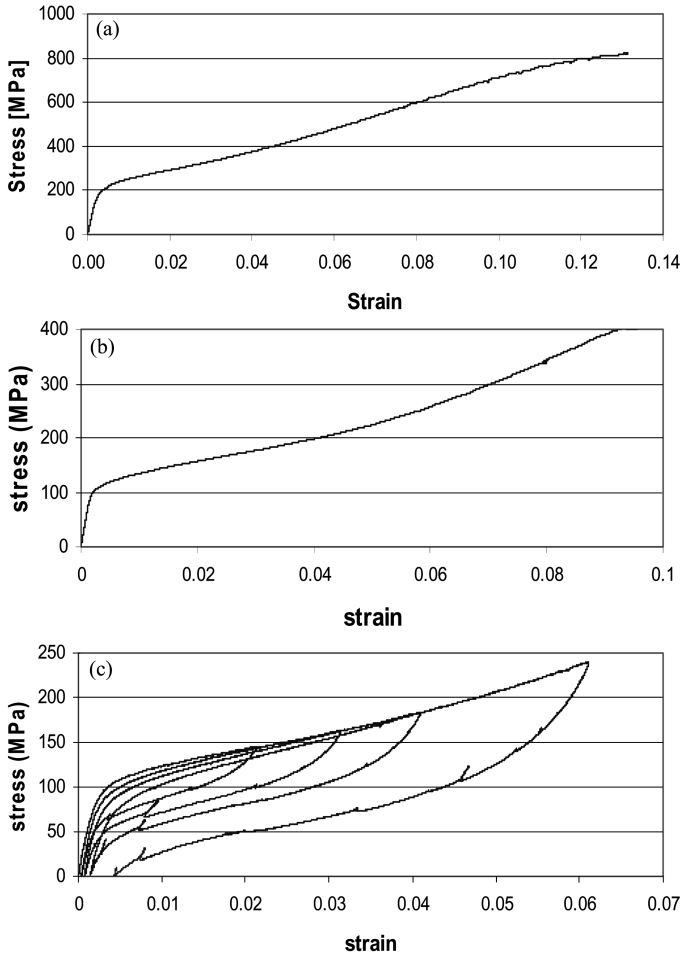


Fig. 20 Tension test for alloy specimens of diameter 3.5 mm coming from the of second producer: up to rupture untreated (a) and treated (b) and with loading-unloading cycles (c)

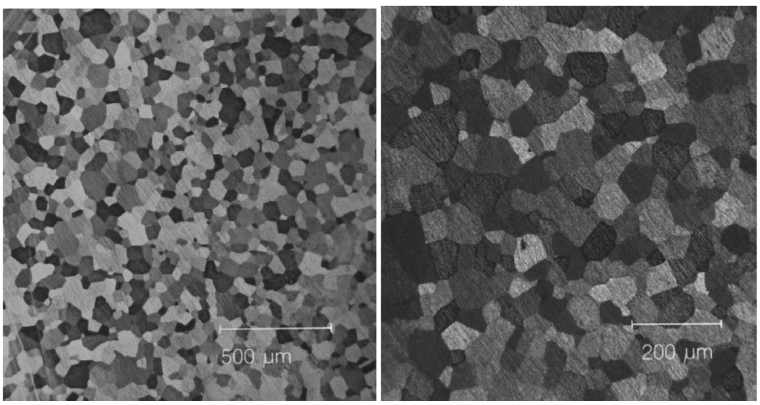


Fig. 21 Optical micrograph - Alloy M

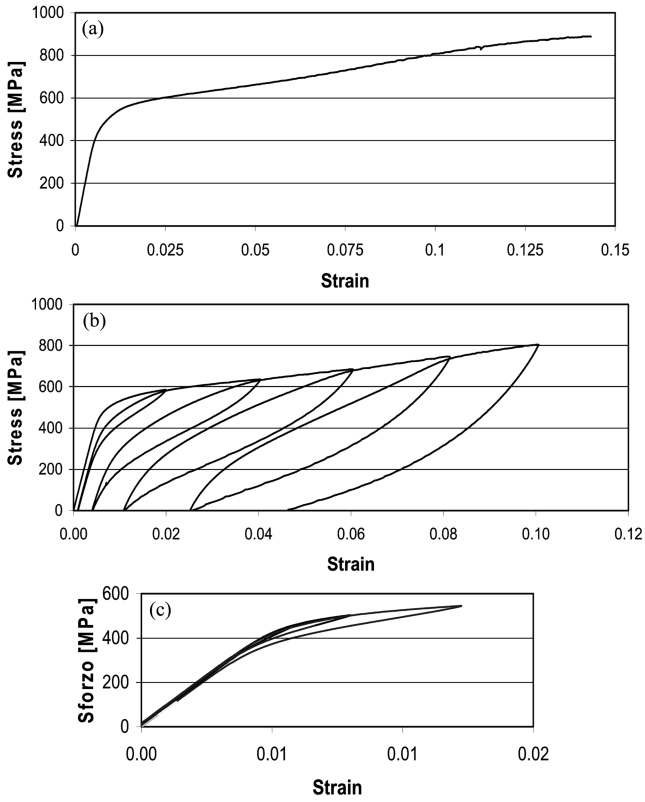


Fig. 22 Untreated alloy with very low martensite transformation temperatures: tension test up to rupture (a), tension test with loading-unloading cycles (b) and repeated cycles at low strain (c)

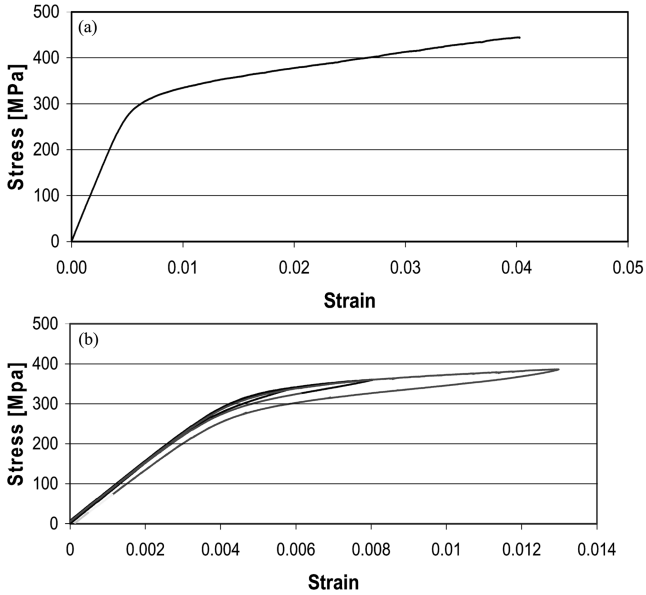


Fig. 23 Treated alloy with very low martensite transformation temperatures: tension test up to rupture (a), tension test with loading-unloading cycles at ambient temperature (b)

coming from the DSC test are once again reported in Table 5. Fig. 21 gives the usual microscopy images and Figs. 22 and 23 collect some results of tension tests in the untreated and treated case, respectively.

## 7. Conclusions

The main contribution of this paper arises from the opportunity the authors had to manage samples of the same Copper-based shape memory alloy coming from different stocks.

Rather similar austenite transformation temperatures were giving the expectation for similar properties. The results collected in this paper show the role of the different thermal and mechanical treatments. But the great influence of the martensite transformation temperatures on the mechanical behaviour of the alloy in its austenite phase is also emphasized.

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