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EFFECT OF ANNEALING TEMPERATURE ON THE MICROSTRUCTURE AND HARDNESS OF Fe-Mn-Si-Cr-Ni-Co SHAPE MEMORY ALLOY PROCESSED BY WIRE DRAWING[1]

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Abstract
Stainless shape memory alloys (SMA's) have been studied since the 90's. The Fe-Mn-Si-Cr-Ni-Co SMA used in this work was processed by wire drawing varying the area reductions, AR's (14%, 39% and 57%) and annealing temperature from 350°C to 1050°C during 1 hour. The microstructural characterization was performed by optical microscopy (OM), scanning electron microscopy (SEM) and Vickers hardness testing. The results showed that the increase of area reduction promoted increase in hardness for the cold drawn and annealed conditions. Results show that the precipitation became noticeable at 450°C and 550°C for samples with AR=39% and 57%, respectively.
Key words: Stainless SMA; Wire drawing; Annealing; Hardness testing.

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1 INTRODUCTION
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According to Wen et al.[1] Fe-Mn-Si shape memory alloys (SMA) have received a special attention in the past 30 years since the discovery of the shape memory effect (SME) in 1982 by Sato. In the 90's they were started the development of stainless SMA as an alternative to the Ti-Ni SMA's due to the low cost, good corrosion resistance, good workability and good weldability.[2-7] The shape memory effect (SME) in these alloys is related to the martensitic transformation γ(FCC)$\leftrightarrow\varepsilon$(HCP),[8-12] which is affected by some factors, such as the alloying elements,[1,2,5,8] annealing treatment[3,8,13] deformation temperature, pre-strain level and thermomechanical training.[1-3,5,8,13] Among these parameters, the annealing treatment is the more effective in the control of SME in Fe-Mn-Si-Cr-Ni based alloys.[8] Some variations in annealing conditions promote microstructural changes in the parent phase, influencing the SME.

The SME is also improved by the presence of some defects, such as Shockley partial dislocations, stacking faults and grain boundaries. These defects function as a nucleation site for $\gamma\rightarrow\varepsilon$ martensitic transformation.[8,13] It has been shown that the increase in the annealing temperature increases the grain size leading to the degradation of SME. This occurs because the larger the grain size, larger will be the distance that the Shockley partial dislocation should move through from one boundary to another, executing the forward and backward $\gamma\leftrightarrow\varepsilon$ transformation.[5,10] When the grain size is small this distance becomes smaller making the $\gamma\leftrightarrow\varepsilon$ transformation easier and therefore the SME is improved.

Various deformation techniques have been applied to produce grain refinement in the microstructure such as forging, rolling and cold drawing.[1,11,14] The degree of area reduction is an effective parameter in the control of the nucleation rate and grain growth during the annealing step.[15,16] The aim of this paper is to evaluate the influence of area reductions in the microstructural evolution and hardness during the annealing in different temperatures of the Fe-Mn-Si-Cr-Ni-Co stainless SMA processed by wire drawing.

3 EXPERIMENTAL PROCEDURES

The chemical composition of Fe-Mn-Si-Cr-Ni-Co stainless SMA used in this work is shown in Table 1. The starting vacuum induction melted ingot of 65 x 65 mm^2 was hot forged down to 20 x 20 mm^2 and then hot rolled down to 6.75 x 6.75 mm^2 corner rounded bar. Then the bar was cold drawn to 2.38 mm intercalating continuous annealing step of 1050°C. Finally the wire was solution treated at 1050°C during 30 min and then water quenched to room temperature. The ingot fabrication and thermomechanical processing up to this stage can be seen elsewhere.[17] The 2.38 mm in diameter wire was cold drawn to a final diameter of 1.56 mm adjusting the intermediate annealing at 2.004 mm (AR=39%), and 1.704 mm (AR=14%). The 57% area reduction wire was obtained drawing directly from 2.38mm wire.

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Table 1. Chemical composition (% wt.) of stainless SMA

Fe	Mn	Si	Cr	Ni	Co	C	S	P
balance	7.79	5.11	13.04	5.90	11.90	0.044	<0.003	0.007

The wires with different area reductions were annealed in a box furnace at temperatures varying from 350°C to 1050°C during 1 hour and then cooled in air. The microstructural characterization of solution treated wire, cold drawn wires and annealed wires were done using a Carl Zeiss optical microscope (OM) coupled with CCD camera and FEI Quanta 400 scanning electronic microscope (SEM). Vickers hardness testing was performed in the longitudinal section of the specimens using a FM-700 microindenter with setting the load to 200gf during10s.

4 RESULTS
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The microstructure of solution treated sample at 2.38 mm is shown in Figure 1.

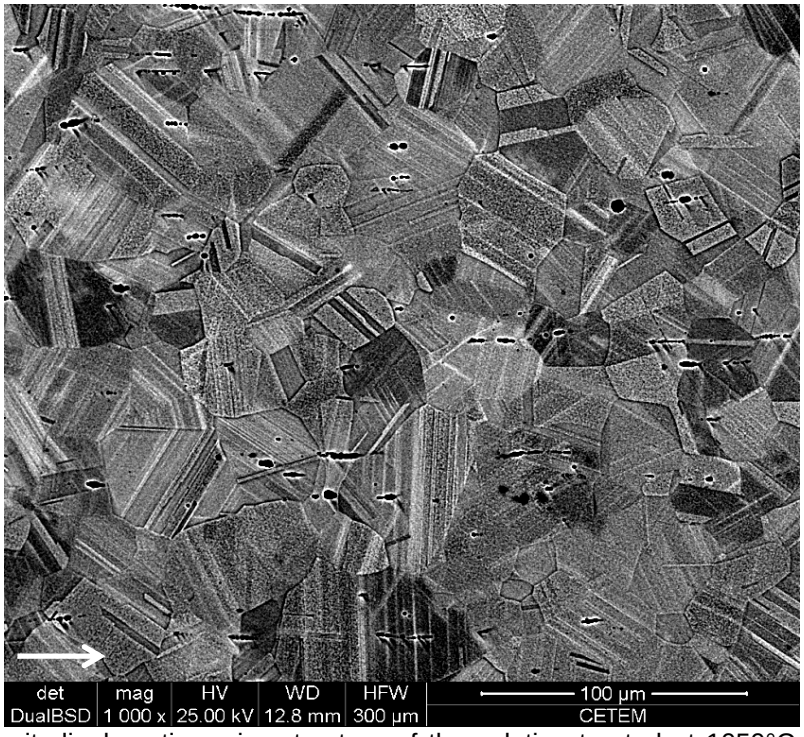

Figure 1. Longitudinal section microstructure of the solution treated at 1050°C during 30 min and 2.38 mm in diameter wire. (SEM-BSE). The white arrow indicates the wire drawing direction.

The microstructure of as drawn conditions wires with area reductions of 14%, 39% and 57%, and annealed conditions at 450°C, 550°C, 750°C, 850°C and 950°C during 1 hour are shown in Figure 2, Figure 3 and Figure 4 respectively.

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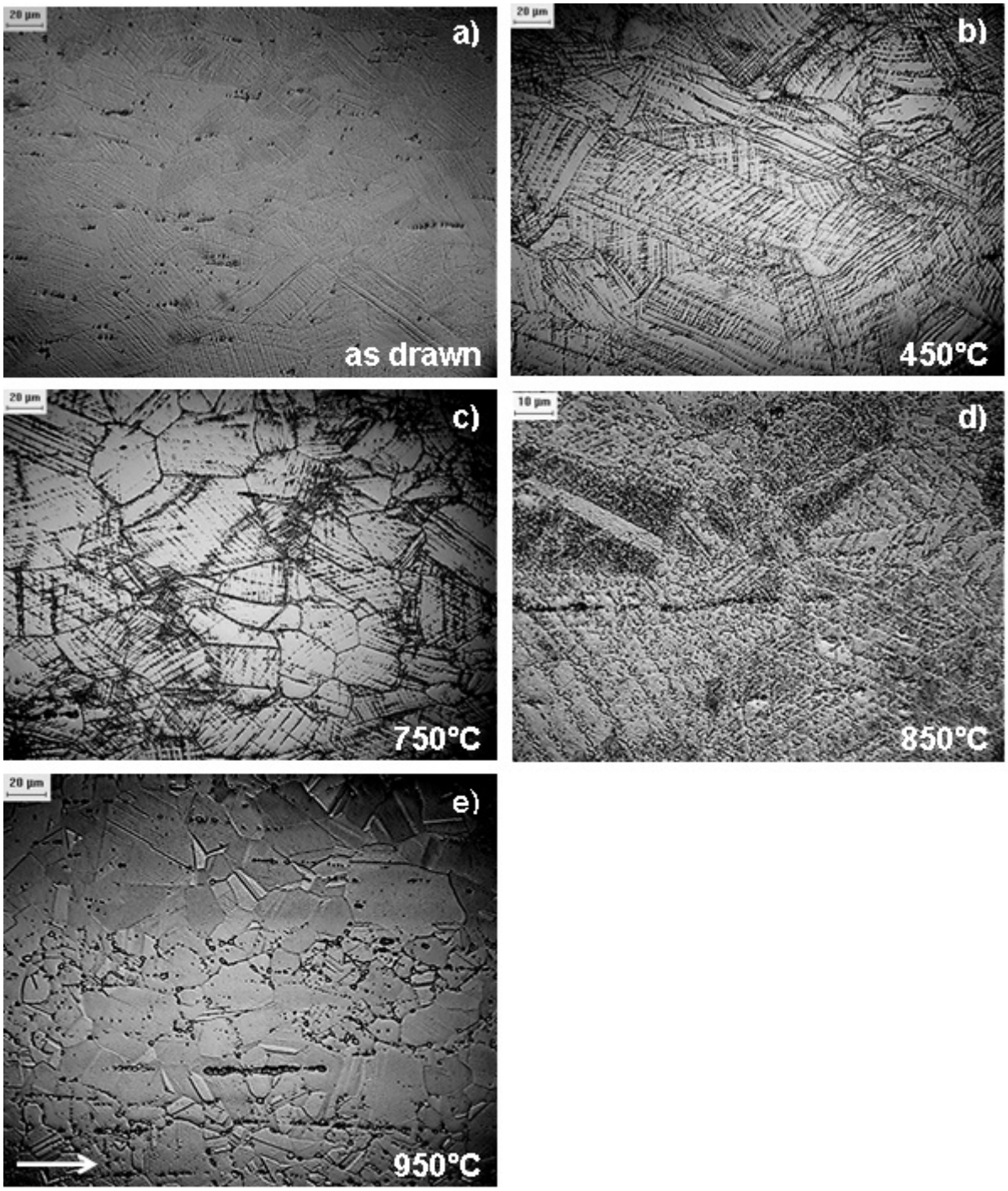

Figure 2. Microstructure of the wire samples with area reduction of 14% in different conditions: (a) as drawn, annealed at (b) 450°C, (c) 750°C, (d) 850°C and (e) 950°C. The white arrow indicates the wire drawing direction.

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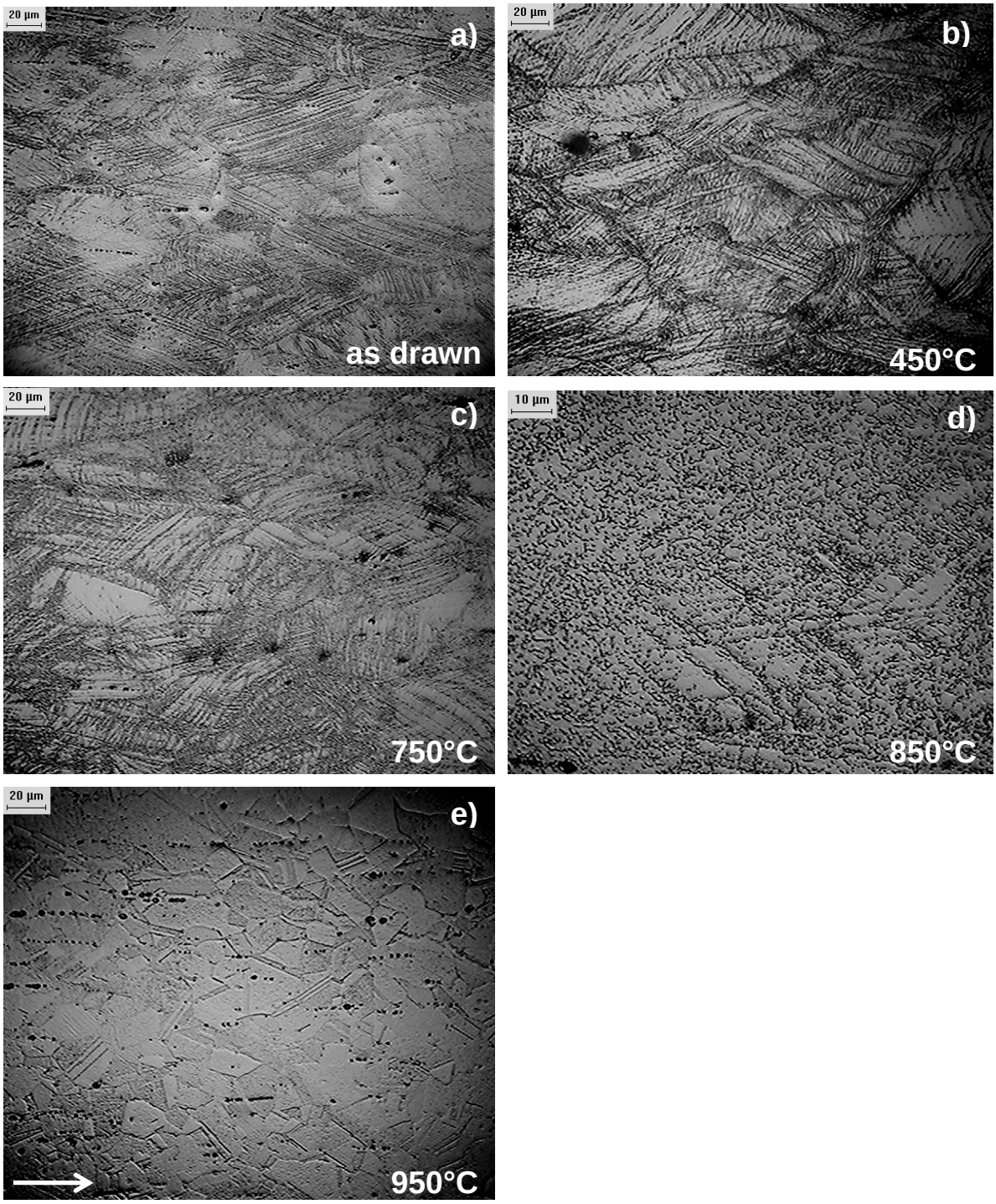

Figure 3. Microstructure of the wire samples with area reduction of 39% in different conditions: (a) as drawn, annealed at (b) 450°C, (c) 750°C, (d) 850°C and (e) 950°C. The white arrow indicates the wire drawing direction.

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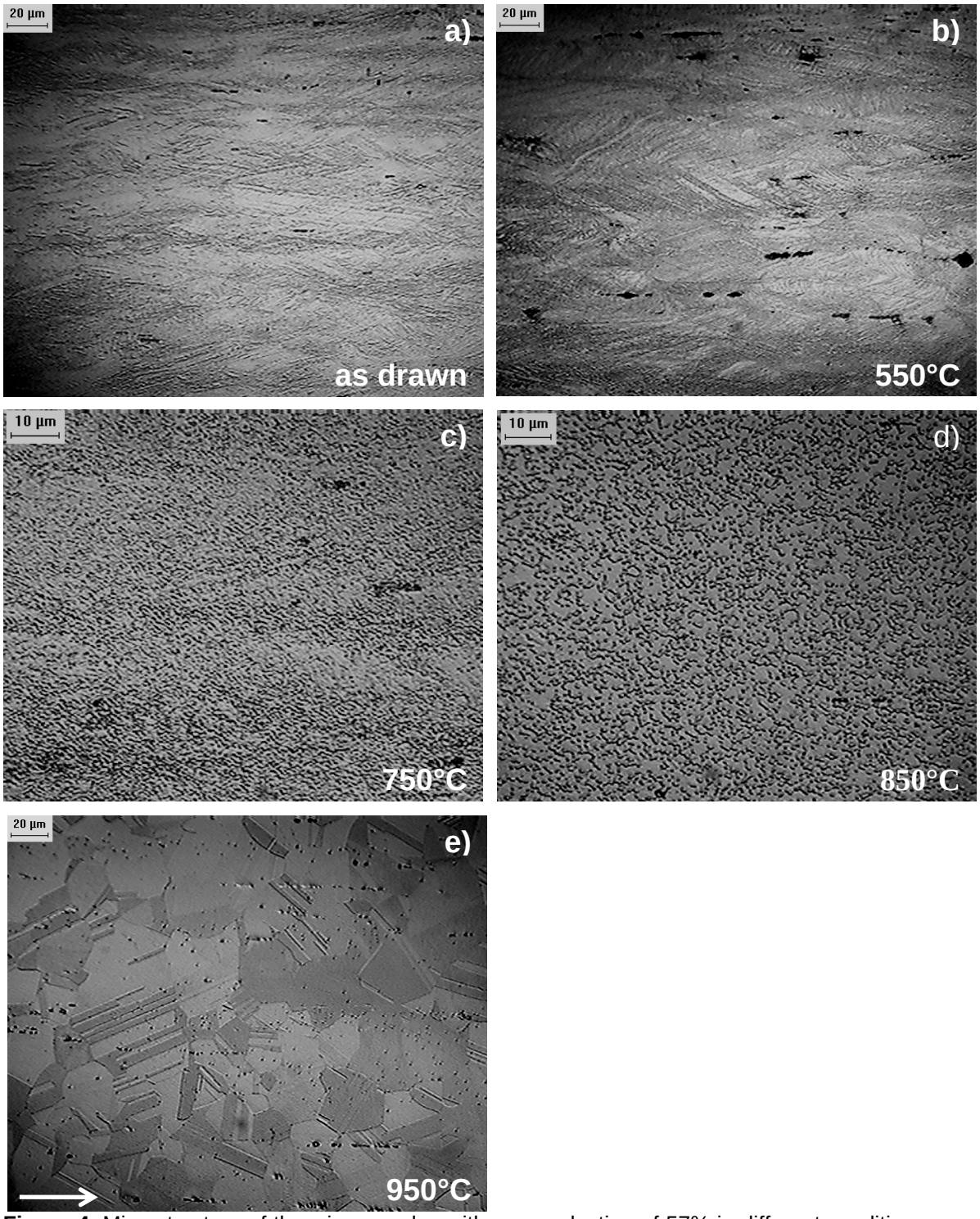

Figure 4. Microstructure of the wire samples with area reduction of 57% in different conditions: (a) as drawn, annealed at (b) 550°C, (c) 750°C, (d) 850°C and (e) 950°C. The white arrow indicates the wire drawing direction.

Figure 5 presents the results of hardness values for the samples in as drawn condition and after annealing in different temperatures from 350°C to 1050°C. For comparison purpose it is shown the hardness values of solution treated samples and then annealed in the same condition of as drawn samples.

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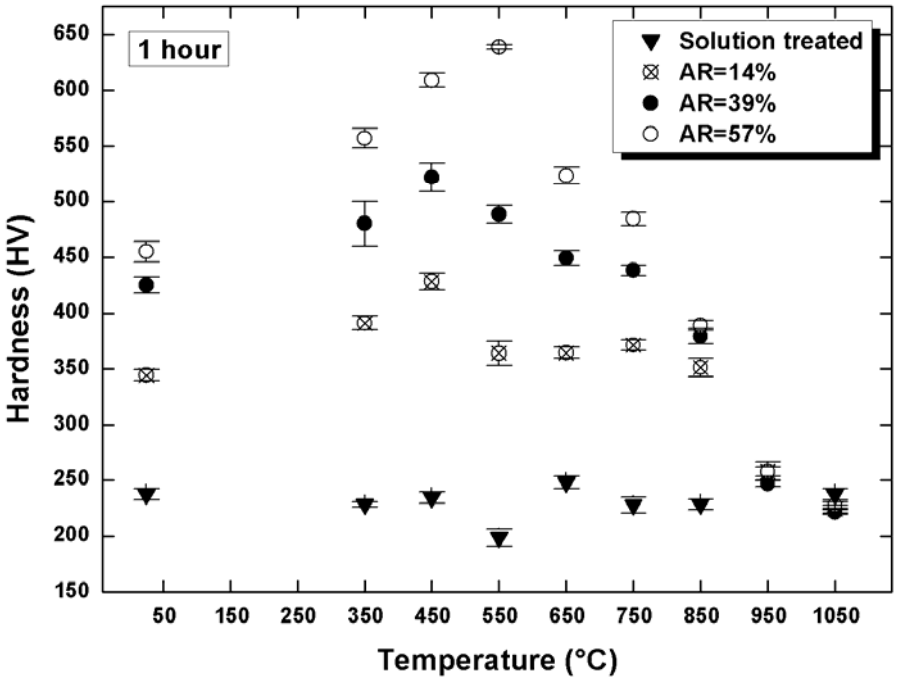

Figure 5. Effect of annealing temperature on the hardness of the Fe-Mn-Si-Cr-Ni-Co alloy wires subjected to 14%, 39% and 57% area reduction respectively.

6 DISCUSSION

It is observed in the microstructure of the solution treated sample (Figure 1) the presence of annealing twins, which are characteristic defects of Fe-Mn-Si based SMA's, that have a low stacking fault energy (SFE).[11,13,18] The effects of increasing the area reductions for as drawn wires are shown in Figure 2a, Figure 3a and Figure 4a. The texture alignment in the drawing directions (horizontal direction of the pictures) increases as the area reduction increases. In Figure 2a one can still see the slip bands and grain boundaries but not in Figure 4a. The Figure 3a shows heterogeneous deformation probably related to favorable preferential orientation on the drawing direction. The cumulative work hardening seen in the microstructure of Figures 2a, 3a and 4a is corroborated with hardness increase shown in Figure 5. The hardness increase from about 237 HV of solution treated sample to a maximum of 455 HV for AR=57%, that is almost twice. It is well known that the larger the cold work more and more crystallographic defects are created in microstructure (i.e. grain boundaries, dislocations tangles, stacking faults and vacancy clusters); which increase the internal energy, raising the strength of material.

As seen in Figure 5, the solution treated sample does not present hardening effect after heat treatment from 350°C to 1050°C compared to drawn wires with variable area reduction. All the drawn wires had their hardness increased after heat treatment being the effect larger the larger is the area reductions. All the samples presented a maximum in hardness being 428HV at 450°C, 522HV at 450°C and 639HV at 550°C respectively for AR=14%, AR=39% and AR=57%. These hardness values higher than that of as drawn

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conditions indicates that the aging process is occurring during posterior heat treatment. Higher maximum hardness values for higher area reduction wires means that more nuclei to promote precipitation are created for larger reduction. The respective microstructures of those wires are shown in Figures 2b, 3b and 4b and one can still see the deformed band structure but not the perception of these nucleated particles. The particles precipitation can be seen in samples annealed at 750°C and 850°C being coarser for the second. That is, precipitates coalescence is occurring.

The softening of the wires samples start at 550°C for AR=14% and 39% and at 650°C for AR=57% as seen in Figure 5. This occurrence could be explained in function of the decrease of crystallographic defects and coalescence of precipitates. The Figures 2c, 3c and 4c show well this effect, but there is still deformation bands for AR=14% and 39% compared with AR=57%. In this stage still is occurring the recovery process of the drawn wires because the annealing at low temperatures is not enough to eliminate completely the complex defects created by cold work. The annealing at 850°C marks the beginning of the recrystallization for the three area reductions, reflecting in a large decrease of hardness. From this point the hardness can decrease to about 50%. The particles precipitation are seen in samples annealed at 750°C and 850°C being coarser for the second as seen in Figures 3d and 4d but less in Figure 2d for lower area reduction. The annealing at 950ºC promoted the dissolution of practically all particles and microstructure are fully recrystallized as seen in Figure 2(e), Figure 3(e) and Figure 4(e) returning to solution treated condition as shown in Figure1. As seen in Figure 2e, the microstructure is not homogeneous presenting variation in grain size which is attributed to a low area reduction.

The literature[1-3,8] reports that the precipitation of second phase particles in Fe-Mn-Si based alloys can improve the SME due to the partitioning effect subdividing the austenite grain in smaller domains and therefore facilitating the forward and backward movement of $\gamma \leftrightarrow \varepsilon$ transformation. In this paper it was not investigated the influence of these particles in the shape recovery but some preliminary results indicate that the annealing at 850°C for AR=57% wire gives the best result in terms of shape recovery. Experiments are underway in order to optimize the thermomechanical processing aiming good shape memory effect and also to identify the particles that appear during pos drawing heat treatment.

7 CONCLUSION

From this work some concluding remarks are:

(1) The solution treated sample presents annealing twins which are characteristic of Fe-Mn-Si based SMA's, that have a low stacking fault energy.

(2) The hardness increased from about 237 HV of solution treated sample to a maximum of 455 HV for AR=57%, that is almost twice indicating the cumulative effect of work hardening.

(3) The solution treated sample did not present the hardening effect but the drawn wires had their hardness increased after heat being their maximum of 428HV at 450°C, 522HV at 450°C and 639HV at 550°C

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respectively for AR=14%, AR=39% and AR=57%. The hardness increase was attributed to a precipitation of second phase particles.

(4) The hardness drecreased as the heat treatment increased and the second phase particles coalesce and finally at 950°C disappear and all the drawn samples returned to solution treated condition.

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