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Microstructuralevolution and mechanical behavior during annealing of aluminum deformed by equal channel angular pressing

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Abstract: This study aims to characterize the microstructure and the evolution of the mechanical behavior after annealing of the AA1070 aluminum alloy cold pressed equal angular channels. The deformation was conducted via route A in five consecutive passes and the accumulated deformation was 5.95. The annealing was carried out at 200 ° C and 250 ° C for 5, 10, 15, 20, 25, 30, 45, 60 minutes. The starting material was derived from a slab of 610 mm thick chopped in multiple passes through hot rolling to final thickness of 32 mm. The microstructure of the material was characterized with the aid of scanning electron microscopy (SEM) in the secondary electron mode. Vickers hardness tests were conducted in order to evaluate the mechanical behavior of the material after pressing and along the heat treatment. The final gran sizes were 1.91µm and 2.07µm ate 200ºC and 250ºC, respectively. The microstructure morphology evolved to near equiaxial. After five consecutive passes of ECAP deformation the average hardness measured was 48.2. The final hardness values after annealing at 200 ° C and 250 ° C were 44.7 Vickers and 40.1Vickers. The reduction at the end of the heat treatment was 7% for the temperature of 200 ° C and 14% for the temperature of 250 ° C. The softening throughout the heat treatment times was due to the activation of the recovery and recrystallization mechanisms that led to changes in the microstructure in order to form a lower energy configuration. The analysis of hardness maps constructed from the data of the mechanical tests allowed concluding that the thermal treatment lead to a development of homogeneity of the hardness distribution.

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I. INTRODUCTION

The mechanical and physical properties of polycrystalline materials are determined by several factors. Grain size is often the factor that influences it in a significant and dominant way. Thus, controlling grain size is one of the methods to produce materials with desired properties and to improve the existing ones. For this reason, ultrafine materials have been of great interest to the scientific community in recent decades [1,2].Severe plastic deformation (DPS) has shown to be promising for this purpose. SPD can be described as a metal forming technique in which the granular structure of the material is refined by the introduction of large plastic deformations. The equal channel angular pressing (ECAP) is considered by the literature as one of the most efficient SPD techniques [3].

Many researchers are currently focusing their efforts on elucidating the mechanisms responsible for grain refinement as well as evaluating the evolution of the anisotropic properties of severely deformed materials at room temperature. Consequently, there are few studies in the literature on thermal stability and the behavior of these materials against annealing [4].When a metal is severely deformed there is an increase in the amount of the stored energy. This increase is associated with the amount of crystalline defects generated in the microstructure during the deformation process.Under these conditions, the microstructure is thermal and mechanically unstable. And it can be easily modified by the dynamic recovery and dynamic recrystallization phenomena, in case of the material is subjected to a critical load even at room temperature. This condition is one of the most critical problems for the practical engineering applications of severely deformed materials and the annealing treatment has proved to be efficient in reducing these instabilities. However, it is difficult reducing instabilities and maintaining excellent mechanical properties at the same time. Thus, studies have been devoted to establishthe adequate annealing time and temperature [5].

In this context, this study analyzed the evolution of the mechanical behavior of the aluminum alloy AA1070 deformed in ECAP via route A and annealed at 200ºC and 250ºC. The microstructural characterization was carried out by MEV and EBSD.The mechanical behavior and its evolution were evaluated by Vickers hardness.

II. MATERIAL AND METHODS

A hot rolled aluminum alloy AA1070 was used in this workwith chemical composition of 0.07 Si, 0.002 Pb, 0.18 Fe, 0.02 Ti, 0.001 Ga, 99.72 Al. A 610 mm thick plate was continuous casted and hot rolled to a 32 mm final thickness. The hot rolling was performed at temperature of 380ºC.The material was pressed in equal channels in five passes in a two-part die of tool steel H13 with two identical perpendicular channels of 10 x 10 mm forming and a radius of the 5 mm channels (R ≈ 37º). The channel and the specimen were lubricated with calcium-sulphonate based grease. The test took place with the speed reaching until 5 mm/s through the route A at ambient temperature. The accumulated real plastic deformation (ε5) was of 5.95.Following, the samples were annealed in a tube furnace at 200ºC and 250ºC during 5, 10, 15, 20, 25, 30, 45 and 60 minutes without atmosphere and cooling rate control.The microstructural characterization was carried out by scanning electron microscopy (SEM). The grain sizes were measured with the aid of the Electron BrackscatterDifraction (EBSD) technique. The Vickers hardness test was executed in the cross-section area of each sample in a Shimadzu Micro Hardness Tester (HVM-2T). The test force was 100 gf during 30s.

III. RESULT

Figure no 1 shows the microstructure of Al AA1070 as received and after five consecutive deformation pass in equal angular channels.

a) **b)**

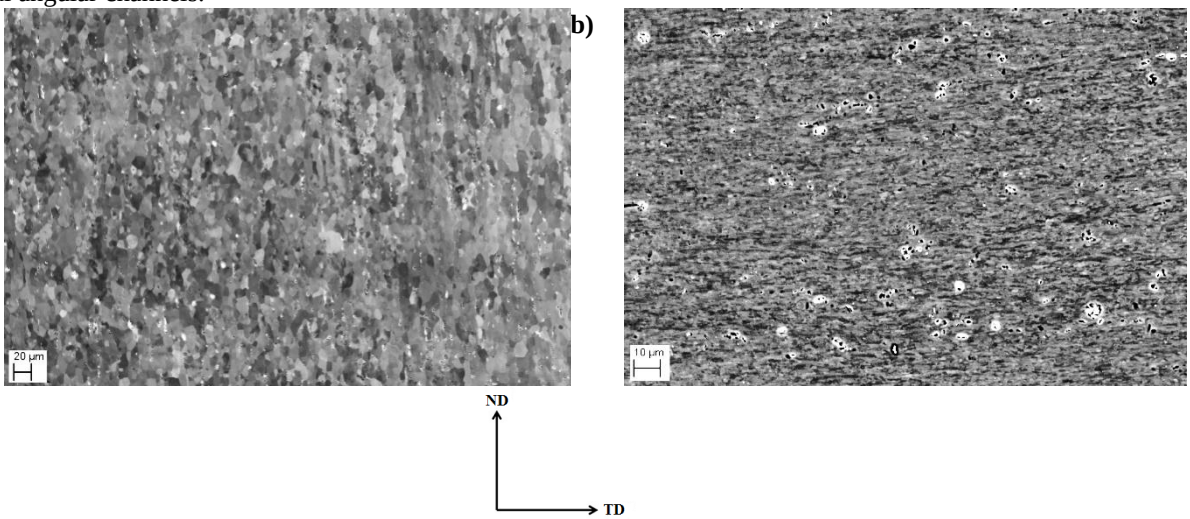


Figure no 1: a) Starting material (Mag: 150); b) Al AA1070 subjected to 5 pressings using route A (Mag: 1500). (MEV – EHT: 10 kV)

Figures no 2 and no 3show the microstructural evolution of annealed material at 200 °C and 250 ºC, respectively

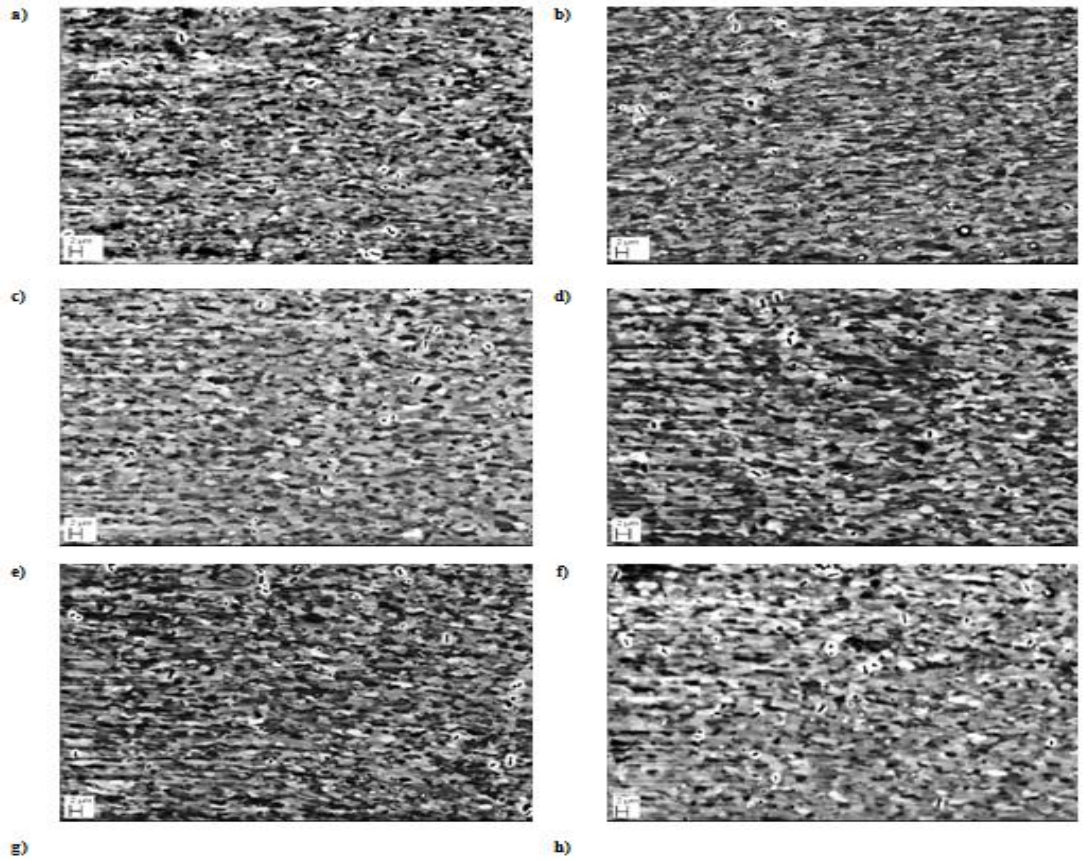
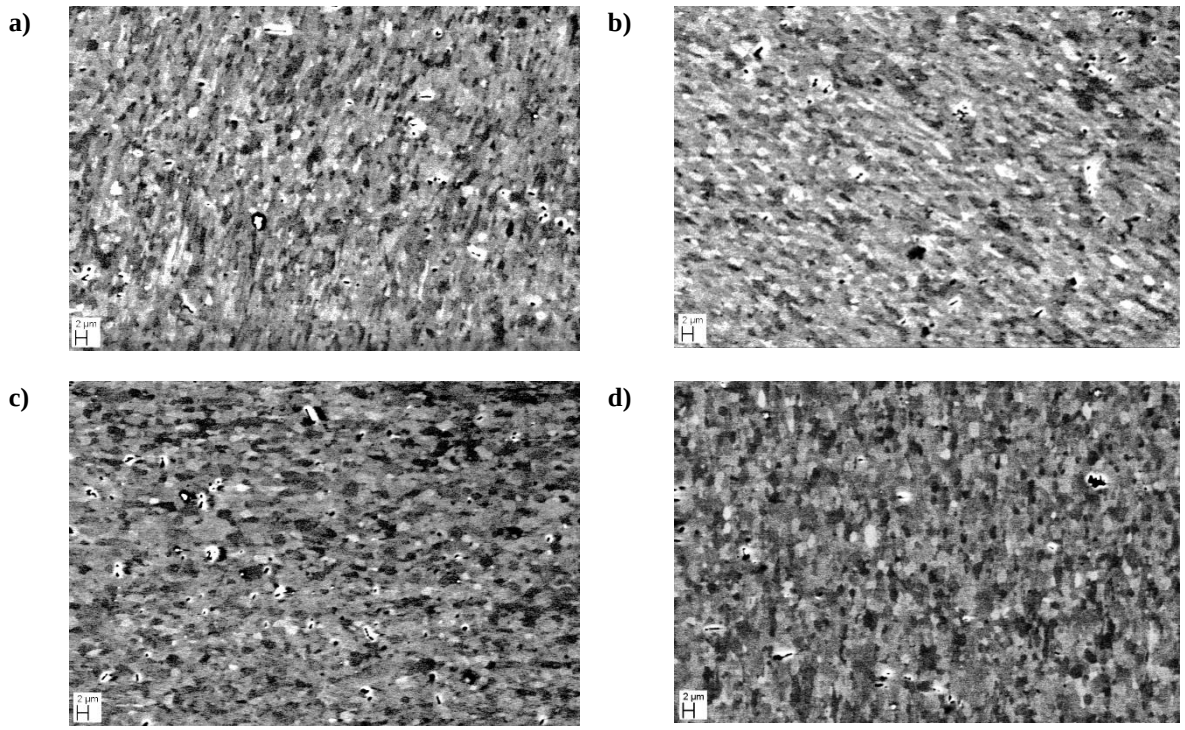


Figure no 2: Microstructural evolution of Al AA1070 deformed by ECAP via route A after 200ºC annealing during (a) 5 min, (b) 10 min, (c) 15 min, (d) 20 min, (e) 25 min, (f) 30 min, (g) 45 min, (h) 60 min. (MEV – EHT: 10Kv – Mag: 3000).



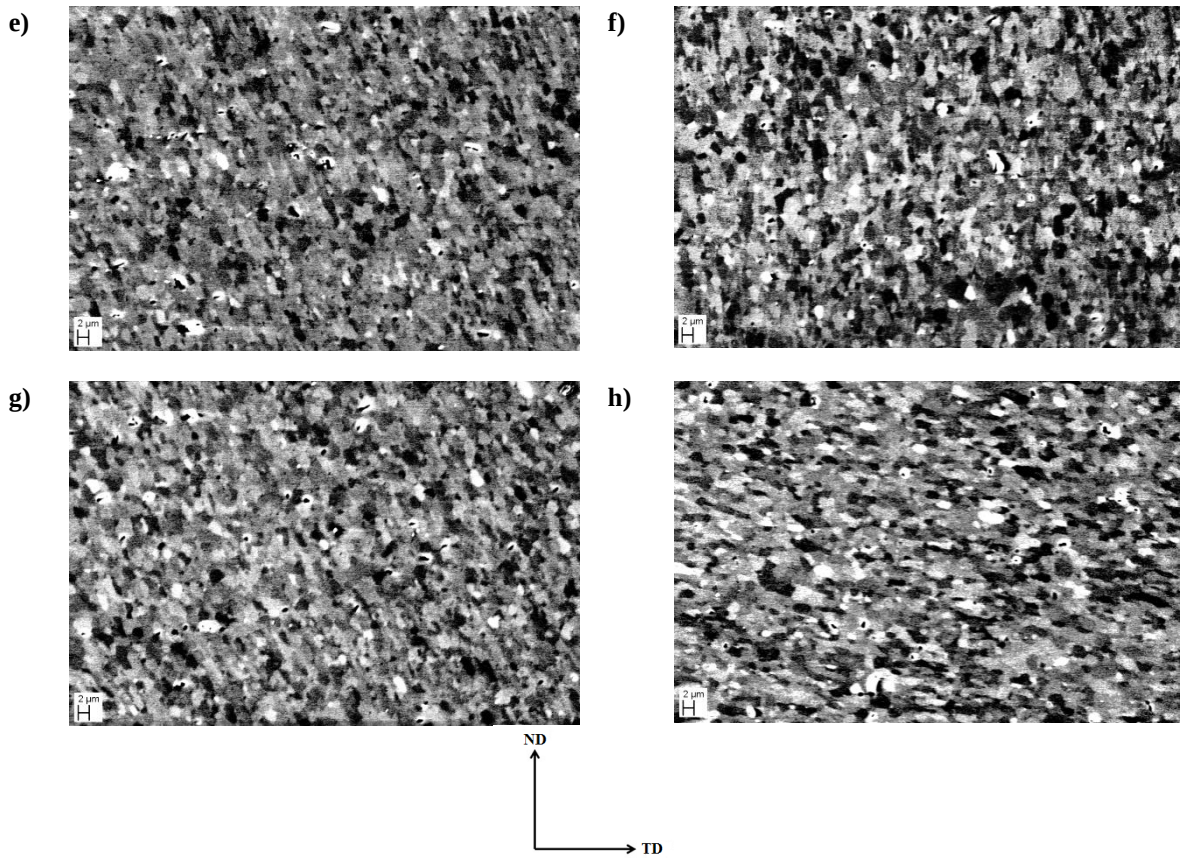


Figure no 3: Microstructural evolution of Al AA1070 deformed by ECAP via route A after 250ºC annealing during (a) 5 min, (b) 10 min, (c) 15 min, (d) 20 min, (e) 25 min, (f) 30 min, (g) 45 min, (h) 60 min. (MEV – EHT: 10Kv – Mag: 3000).

Table no 1 and Figure no 4 show the grain size evolution of the material after annealing at 200ºC and 250ºC.

Table no 1: Grain size of the Al AA1070 after annealing.

Annealing time (min)	Temperature	
	200 ºC	250 ºC
	Grain size (μm)	
5	1.77 ± 0.70	1.97 ± 0.75
10	1.81 ± 0.69	1.98 ± 0.73
15	1.96 ± 0.81	1.99 ± 0.76
20	1.88 ± 0.70	2.00 ± 0.82
25	1.89 ± 0.70	2.04 ± 0.79
30	1. 88 ± 0.70	2.05 ± 0.82
45	1.89 ± 0.70	2.04 ± 0.79
60	1.91 ± 0.65	2.07 ± 0.82

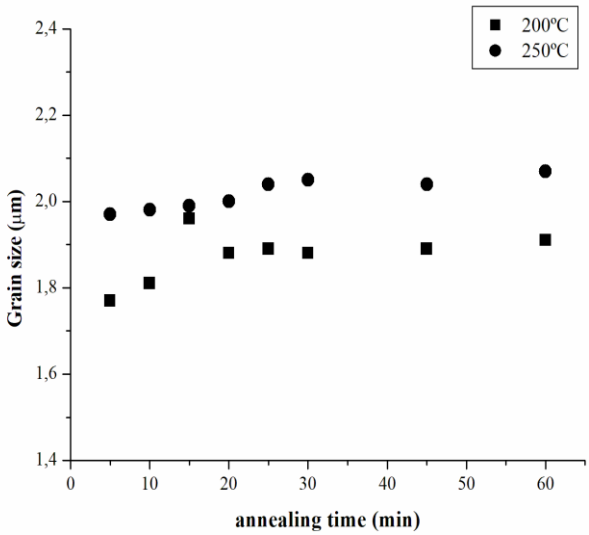


Figure no 4: Grain size evolution of Al AA1070 subjected to ECAP via route A and annealed at 200ºC and 205ºC.

Figure no 5 shows the hardness maps of the as received material and after five passes via ECAP. The hardness values were 25.5 ± 0.8 Vickers and 48.2 ± 1.6 Vickers.

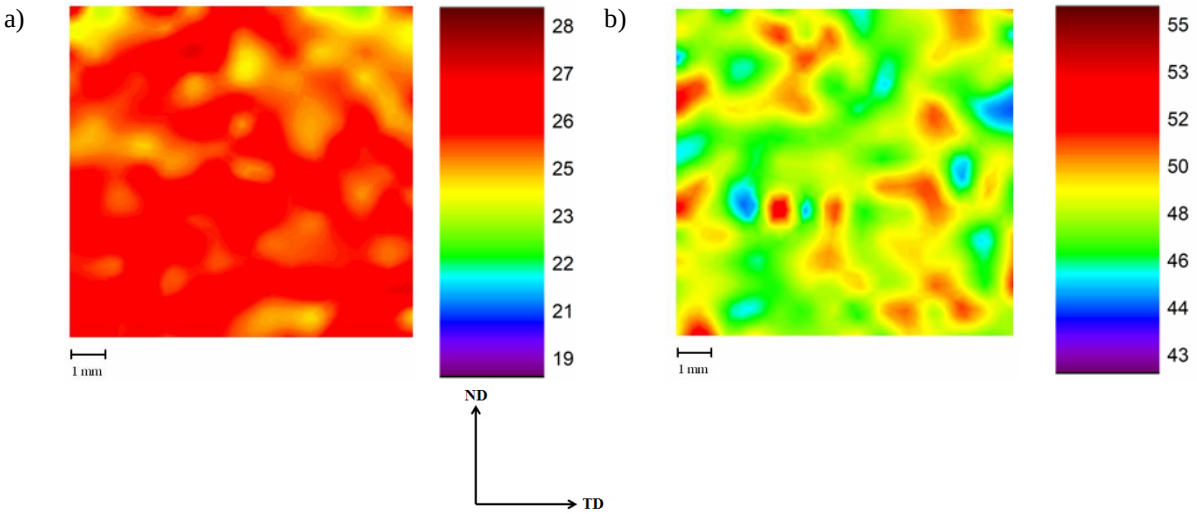


Figure no 5: Vickers hardness maps in the cross-sectional area: (a) starting material (b) sample subjected to 5 pressings using route A.

Table no 2 and Figure no 6 show the Vickers hardness values of the material as a function of time and annealing temperature.

Table no2: Al AA1070 Vickers hardness.

Annealing time (min)	Temperature	
	200 ºC	250 ºC
	Hardness (Vickers)	
5	47.1 ± 1.1	43.4 ± 1.0
10	47.1 ± 0.9	43.5 ± 0.6
15	46.7 ± 0.7	42.6 ± 0.7
20	46.1 ± 0.9	42.2 ± 0.7

25	46.1 ± 0.8	41.9 ± 0.7
30	45.7 ± 0.6	40.5 ± 1.3
45	44.1 ± 1.3	40.7 ± 0.9
60	44.7 ± 0.8	40.1 ± 0.9

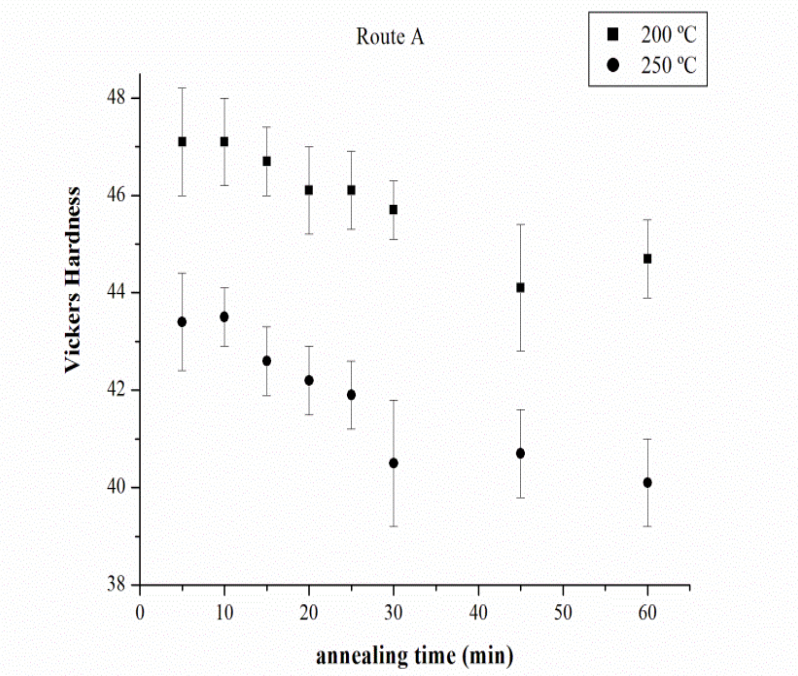


Figure no 6: Al AA1070 deformed by ECAP via route Avickers Hardness evolution during annealed at 200ºC and 250ºC.

Figures no 7 and 8 show the Vickers hardness maps of the material deformed by ECAP via route A Vickers hardness during annealed at 200ºC and 250ºC

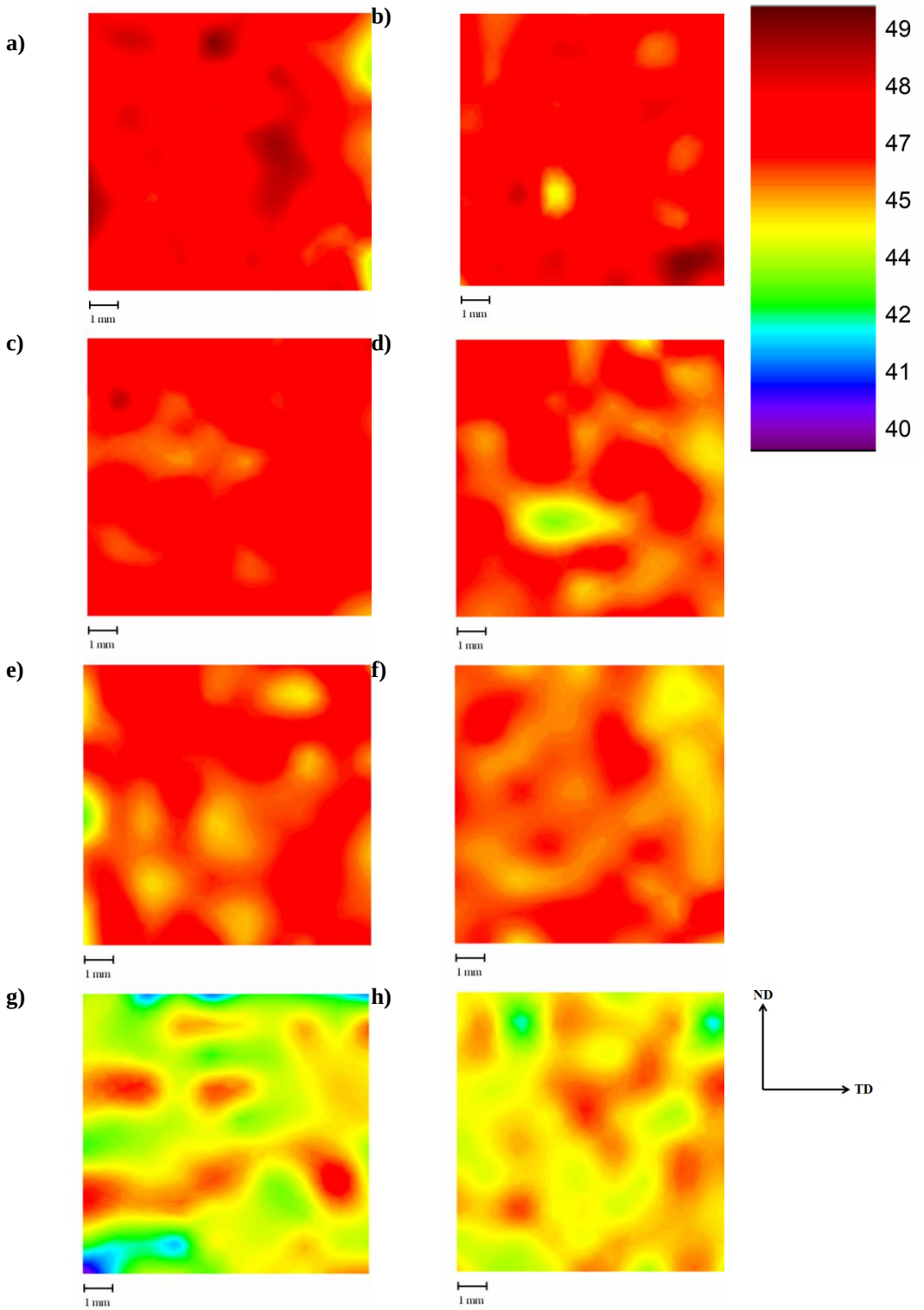


Figure no 7: Vickers hardness maps in the cross-sectional area of Al AA1070 deformed by ECAP via route A after 200ºC annealing during (a) 5 min, (b) 10 min, (c) 15 min, (d) 20 min, (e) 25 min, (f) 30 min, (g) 45 min, (h) 60 min.

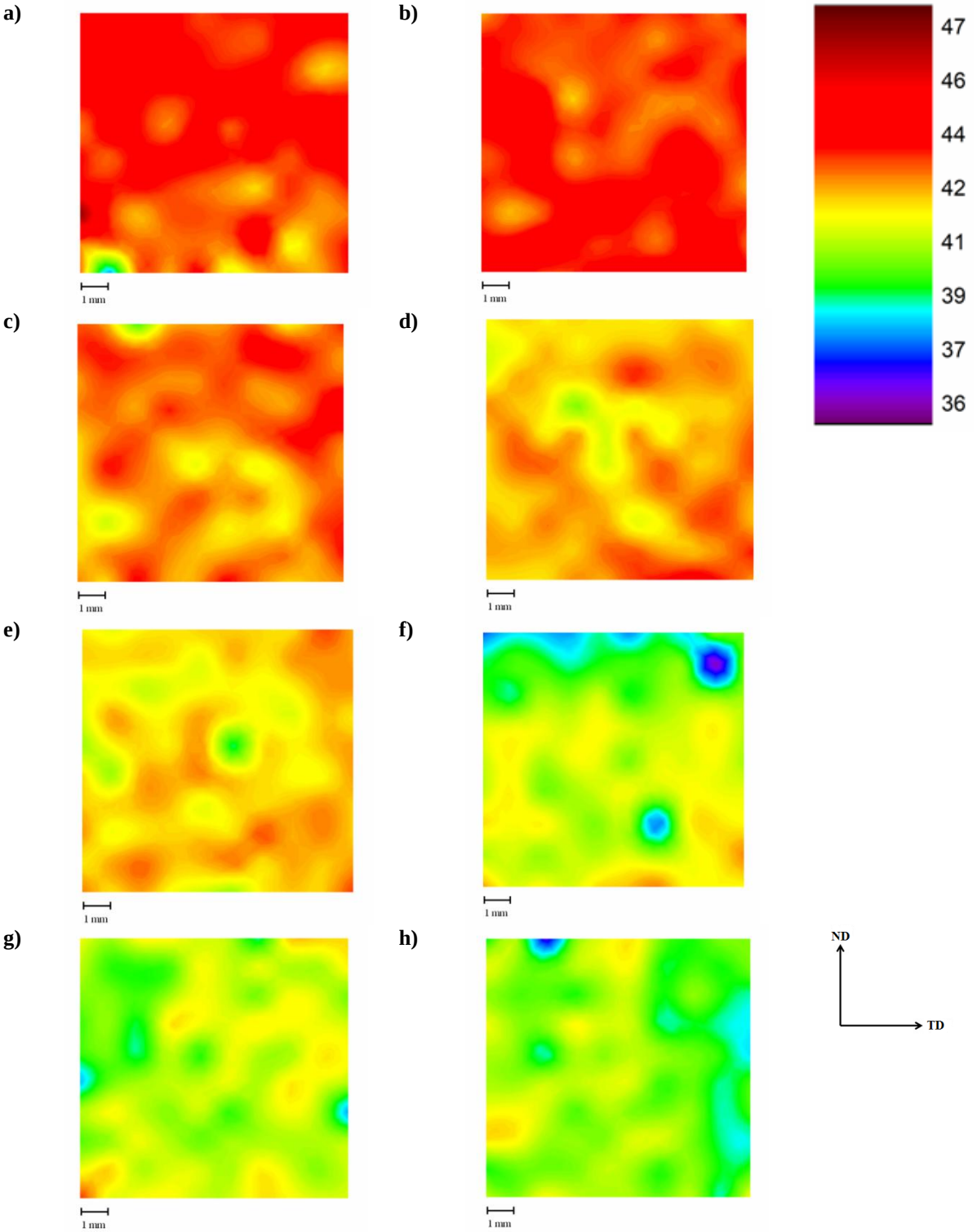


Figure no 8: Vickers hardness maps in the cross-sectional area of Al AA1070 deformed by ECAP via route A after 250ºC annealing during (a) 5 min, (b) 10 min, (c) 15 min, (d) 20 min, (e) 25 min, (f) 30 min, (g) 45 min, (h) 60 min.

IV. DISCUSSION

The cross-sectional micrograph shows a partially recrystallized and heterogeneous microstructure composed of grains with different morphologies and irregular sizes. These characteristics indicate the occurrence of dynamic recovery and dynamic recrystallization phenomena during the hot rolling process. The average grain size was 19.65 μm. It was verified an abnormal growth of grains, this phenomenon is also known as secondary recrystallization and is the common occurrence in hot deformed aluminum alloys.

The fifth pass of deformation via route A resulted in an average grain size of 2.4 μm, an 85% reduction over the starting material grain size. The grains showed an elongated morphologyand a fraction of high angle boundaries of 0.69. These characteristics are foreseen in the literature [6-8].

It was observed in the beginning of the heat treatment at 200ºC the initial structure, consisting of elongated grains, begins to change to an approximately equiaxial structure. And the grain size at the end was 1.91 μm. The high angle boundary fraction after the treatment was 0.80. At the end of the treatment at 250 ºC the grain size was 2.07 μm. This represents just a slight reduction compared to deformed material. The high angle boundary fraction was 0.78. The final values of grain size for both temperatures reduced compared to the deformed material. This behavior is explained by the occurrence of the recrystallization phenomenon, characterized by the growth of a new matrix over the deformed one. The recrystallized grains can be seen in Figures no1 and 2.

The hardness average of the starting material was 25.5 ± 0.8 Vickers. This value and its standard deviation associated with the hardness maps show certain microstructure homogeneity along the cross-section. After five consecutive pass of deformation the hardness value increased47% of the starting material average hardness. This increase is justified by the high imposed strain ($\varepsilon = 5.95$), associated to the increase of crystalline defects in the microstructure and the intense grain refinement.Xu and Langon [9] attribute this behavior to the fact that pure aluminum has high stacking fault energy, a high strain hardening rate and a rapid recovery rate. The increase was found right after the first deformation pass, likely reported by several works [10-13]. It was also showed that there were no significant changes in the hardness values in the following passes considering the standard deviations. This behavior characterizes a steady state justified by the occurrence of the dynamic recovery phenomenon during the ECAP. Thus, the amount of defects generated during the deformation is compensated by the number of eliminated defects by the movement of the dislocations.

The severely deformed materials exhibit thermal instability, despite their improved mechanical properties, due to the high density of crystalline defects. The thermal stability of the processing material can be improved with the return heat treatment. The final values shows a slight reduction of approximately 7% of the hardness averagein relation to the deformed material at the end of the heat treatment at 200 ° C and 17% at 250 ° C. This softening was expected and is in accordance with the literature [5,14-16].The analysis of the hardness maps shows that hardness measurement homogeneity was reached after the heat treatment.

Severe plastic deformation results in a very intrinsically instable microstructure and the evolution of the substructures with the subsequent annealing are conducted by thermally activated processes, reducing the stored energy [17].Therefore, the gradual softening throughout the treatment time is justified by the occurrence of recovery and recrystallization phenomena. Thematerial subjected to ECAP presented a high hardness value due to the intense grain refinement, increase of the fraction of discordances and punctiform defects increasing the stored energy.The softening after annealing is due to the activation of restoration mechanisms that led to changes in the microstructure in order to achieve a lower energy configuration.The main changes are the elimination of punctiform defects, annihilation and rearrangement of opposed signal dislocations, formation of high angle boundaries, a new free dislocation matrix and the growth of these grains consuming the deformed matrix.Huang et al. [18] confirm in their work that the dislocation elimination generated by the annealing and the increase of the density of these defects during deformation processes are the main causes of the changes in the mechanical behavior of the materials.

The low reduction in the hardness values at the end of the heat treatment can be justified by the fact that the grain size has remained practically constant throughout the treatment times. Furthermore, it is known that recrystallization involves the development of a new grain structure with low density of disagreement, through the formation and migration of high angle contours [19]. Also, it occurs by nucleation and grain growth. Nucleation corresponds to the appearance of new grains. During growth, these new grains take the place of the deformed microstructure. That is, it can be concluded that the new grains resulting from the recrystallization did not grow significantly and kept their sizes close to the grains of the deformed matrix.

The literature points out that it is difficult to reduce instabilities and maintain excellent mechanical properties at the same time. However, the results obtained in the present work are satisfactory since the intention of applying the ECAP technique to obtain a refined granular structure with high mechanical strength was reached at the end of the heat treatment providing a thermally stable and equally resistant material.

V. CONCLUSION

The microstructure was characterized and the evolution of the mechanical behavior of the alloy AA1070 pressed in equal angular channels via route A and annealed at 200ºC and 250ºC was evaluated. The grain size showed little variance for both temperature and the microstructure morphology and its evolution were guided by the occurrence of recovery and recrystallization. The average hardness value decreased by 7% after annealing at 200ºC and 14% to the temperature of 250 ° C. The gradual reduction was due to microstructural

changes resulting from the occurrence those phenomena along isothermal annealing. The instabilities were reduced and a refined granular structure material with high mechanical strength was obtained.

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