

## Microstructural Evolution of Nickel-Based Superalloy Processed By Equal-Channel Angular Pressing (ECAP)

Renan Augusto Francisco Dias<sup>1\*</sup>, Waldemar Alfredo Monteiro<sup>1</sup>

<sup>1</sup>Instituto de Pesquisas Energéticas e Nucleares – IPEN, CCTM, SP, Brazil

\*rafd\_rafd@hotmail.com

The present work shows the microstructural (under optical microscopy) and Vickers hardness evolution of a nickel-based superalloy Inconel 600 (alloy 600) before and after deformed by Equal-Channel Angular Pressing (ECAP) that significantly modified the alloy microstructure and, consequently, its strength. Alloy 600 is an austenitic nickel-based superalloy with 72% nickel, 14-17% chromium and 6-10% iron and it is commonly used in structures and components that work in aggressive environments. The grain size plays a significant role in the mechanical properties in this alloy; so, it is important to understand how processing techniques modify the microstructure of the material. In fact, ECAP is a processing technique involving the application of severe plastic deformation (SPD) used in the manufacture of metals and alloys with ultra-fine grains (UFG) and therefore with extraordinary combinations of both high strength and high ductility [1]. For the research, alloy 600 rods, supplied by Multialloy, with 8 mm diameter, were heat treated at 1200 °C for 6 h (solution), as shown in Figs. 1a and 1b, to achieve a larger grain size. The generated products were machined to the final dimensions of the specimen (6 X 6 X 25 mm) shown in Fig. 1c and then processed by ECAP, at room temperature using route A, in a D2 tool steel die, whose angles  $\Phi$  and  $\Psi$  are 120° and 0° (Fig. 1d). Samples were pressed repetitively through a total of three passes, the microstructural aspect of the samples after each pass are shown in Figs. 2 and 3 with different magnifications. Metallographic preparations were made in the transverse, longitudinal and normal directions, TD, LD and ND of each sample. Sanding step with grit 320, 600 and 1200 were used. Polishing steps included 3  $\mu$ m- and 1  $\mu$ m-diamond paste and 0.05  $\mu$ m-alumina suspension and finally they were etched for optical examination using Marble's etchant (10 g CuSO<sub>4</sub>, 50 ml HCl and 50 ml H<sub>2</sub>O). The micrographs were taken with OLYMPUS optical microscope model BX51M, with magnifications of 100x and 200x. The hardness tests were conducted in a Buehler Micromet 2103 microdurometer in the LD of the samples. For each measurement, a load of 500 g was applied for 15 s. Five separate measurements were taken on each sample at randomly selected points and then averaged. In Figs. 1a and 1b, it is possible to see only 1 phase, a solid solution of Ni-Cr-Fe, a coarsened grain structure and annealing twins. There are slight changes in the microstructure after the first pass, some strain marks are seen. After the second and third passes the microstructure shows highly deformed grains, deformation and transition bands. The values of hardness (Tab. 1) show an increase after each pass. In summary, (1) the micrographs analysis shows all the transformations from an annealed state to a highly-deformed state; (2) the grains are yet elongated after 3 passes and possibly do not show an expected UFG structure; (3) deformation twinning is revealed and is possible to see strain marks, deformation and transition bands; (4) the hardness values increased due to the high strain imposed to the alloy.

**References:**

[1] ZHILYAEV, A.P. et al., The microstructural characteristics of ultrafine-grained nickel, *Mat. Sci. and Eng. A* 391 (2005) 377–389. 2004.

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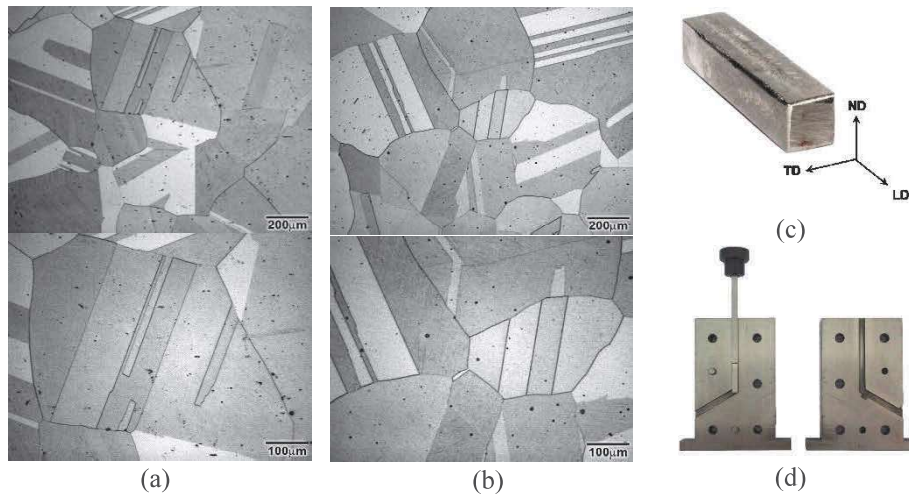


Fig. 1. (a) Microstructure of alloy 600, solution annealed, LD. (b) Microstructure of alloy 600, solution annealed, TD. (c) ECAP Sample before pressing. (d) ECAP die.

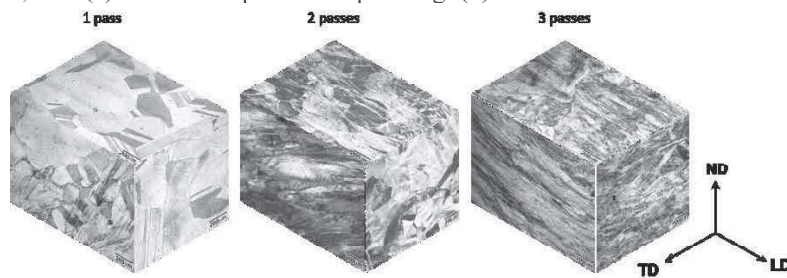


Fig. 2. Microstructure of alloy 600 after each pass. (bar = 200 μm).

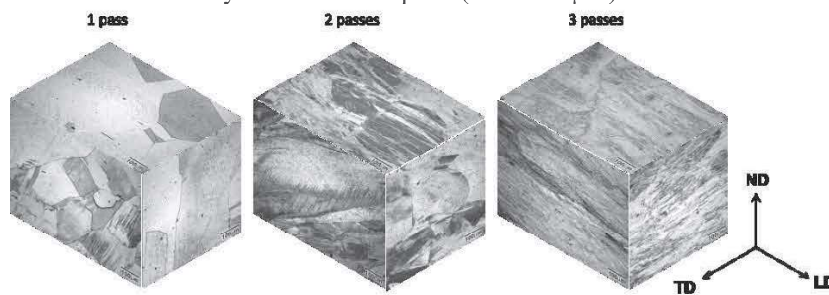


Fig. 3. Microstructure of alloy 600 after each pass. (bar = 100 μm).

Tab. 1 Table of hardness values for the samples

Sample	Hardness Vickers (HV0,5)
As-Received	302 ± 5
Solution	150 ± 7
1 pass	332 ± 14
2 passes	409 ± 15
3 passes	450 ± 18