Journal of Material Sciences & Engineering

Schwartz et al., J Material Sci Eng 2015, 4:6
DOI: 10.4172/2169-0022.1000205

Research Article | Open Access

Failure of an Inconel 718 Die used in Production of Hot Copper Direct Extrusion

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Abstract

Steel dies used in hot extrusion of copper failed after several extrusion cycles (2-5 tons of extruded material). In the extrusion process the die is subjected to high temperatures and stresses the failure mechanisms was plastic deformation. Industry considers new die materials, such as Inconel 718. In this research it was shown that during production using an Inconel 718 die one can extrude 8 times more material (approx. 40 tons). After the extrusion of 40 tons of copper small cracks and plastic deformation on die aperture are seen. The die was discarded and used to investigate die failure mechanisms. By using different investigation methods (optic and scanning electron microscopy, hardness and microhardness tests) microstructure changes were observed. This research was done in the collaboration of Politehnica University of Bucharest, Romania and S.C. LAROMET S.A. (a manufacturer of copper and copper alloys).

Keywords: Hot copper extrusion; Inconel 718 die; Delta phase; Cracking

Introduction

Inconel 718 is a very important material for aerospace applications due to excellent mechanical characteristics in the -253 to 650°C temperature regime. It is a nickel – iron base superalloy with 5.3% Nb content [1].

Solid solution and precipitation strengthening are the strengthening mechanisms. The matrix, denoted gamma phase (γ) is reinforced by two precipitated phases: gamma prime (γ'), which has a FCC structure and is an ordered $Ni_3(Al,Ti)$ phase and gamma double prime (γ''), also a coherent with a BCT structure Ni_3Nb phase. The γ'' phase is metastable. A third and detrimental phase, considered to reduce strain, is the brittle delta phase (δ) which appears as a Widmanstatten structure and can act as a stress raiser. The delta phase is incoherent, with an ortorombic structure and the same chemical composition as the tetragonal γ'' [2].

The δ phase formation occurs in the 650-980°C temperature range with platelet morphology. Recent studies show the beneficial effect of δ phase: it can be used to control grain size and has a pinning effect by blocking grain boundary sliding [3-5].

The most rapid δ phase precipitation was found by Moll at temperature of approximately 900°C, very close to the working temperature regime found in hot extrusion of copper [6].

Microstructure evolution in Inconel 718 in high temperature working regimes are widely available but cannot be applied to conditions found during hot extrusion of copper. In LAROMET S.A. the hot extrusion of copper is performed with a billet temperature approx. 850°C, well above the 650°C limit temperature for Inconel. The high temperature associated with high stresses occuring during extrusion creates a new working regime with unusual parameters for this alloy.

Our aim is to observe and compare the microstructure evolution in this working regime with literature data. Based upon these results further actions of die maintenance, design or even new materials will be taken into account.

Usually steel dies made from AISI H13 were used for copper hot extrusion. These dies failed by severe plastic deformation and needed either refurbishing or even discarding only after an average of 5 tons of extruded copper. This experimental Inconel 718 made die managed 40 tons of extruded copper with little refurbishing, 8 times more than an old steel die.

Materials and Methods

The die was still usable, but it was decided to discard it for research. First the surface was polished for hardness measurements and then samples were obtained using a metalographic cutter for optic and scanning electron microscopes studies, X-Ray analysis and microhardness tests.

Prior all destructive tests a macroscopic observation was performed, by naked eye, to find most relevant regions for further studies. For the hardness mapping on the polished die surface a grid 20 × 20 mm was traced and at each intersection four HRC measurements were performed, whenever possible. The data was recorded and inserted in a commercial software, converted in a matrix and a hardness map was plotted.

For optic and scanning electron microscopy studies the samples followed a standard metallographic preparation as recommended by several authors [7-9].

The XRD patterns are obtained using $Cu_{K\alpha}$ radiation in a conventional difractometer and the microhardness tests are performed using a Vickers indenter and a load of 40 gf.

Results and Discussion

In Figure 1 the regions of interest on the discarded die are depicted. It was a 320 mm diameter die with a rectangular aperture for extrusion.

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Received October 08, 2015; **Accepted** October 24, 2015; **Published** November 04, 2015

Citation: Schwartz M, Gheorghe D, Ciocoiu R, Ciuca I, Jula G (2015) Failure of an Inconel 718 Die used in Production of Hot Copper Direct Extrusion. J Material Sci Eng 4: 205. doi:10.4172/2169-0022.1000205

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After polishing cracks at aperture corners are observed, growing at 45° angles. A significant plastic deformation can be observed at the middle of the aperture length. Since plastic deformation was not a concern, mainly samples from the cracked areas were analyzed.

The initial HRC hardness, on the new die, had an average value of 43 HRC with a 0.2 standard deviation. After use, on the back side a 44HRC mean value was found, but with a standard deviation of 1.2. The back side of the die did not show any damage signs and no further analysis was performed.

On the front side of the die hardness variation is plotted in Figure 2.

The maximum hardness of 42HRC was found near the edge of the die, far from the aperture. Little or no microstructure changes were expected in this region, Sjoberg [10] reporting hardness for heat treated Inconel 718 ranging from 32 to 37HRC and a 42HRC value is normal for forged, annealed 1065°C/1h/air cooled and aged 760°C/20 h [11].

The minimum value was found at 25HRC where a severe dropdown of 17HRC units occurred. Regions near aperture show lower values but in a median region also lower values can be observed. By hardness variation on the surface microstructure change is to be expected. The distribution reveals that the left side of Figure 2 has an overall lower hardness while for the right side appears to have a higher hardness. Such unequal distribution could be obtained by a non uniform loading of the die during extrusion which produces a shear.

Using the optic microscope a general aspect of the main crack was observed. The crack is intergranular, it grows on grain boundary. From the main crack many secondary cracks split and grow on grain boundary and then re-intersect with the main crack, the result is isolated grains or groups of grains, as Figure 3 clearly depicts. Also an uniform grain size is observed; the grains are small and did not coarsen.

The main crack grows at grain boundary and it has a saw-tooth appearance as seen in Figure 4. This crack originates at aperture corner and extends at a 45° angle. At higher magnification, (Figure 5) it can be seen how the crack splits and grows on grain boundary, surrounds a grain or a group of grains and the re-intersects with the main crack.

In Figure 6 a crack which occurred in the proximity of the main crack can be observed. Its growth direction is nearly parallel to the main one, but no contact with the exterior environment was made. In its path the crack interacts with carbides, as shown in Figure 7. The crack is deflected and forced to grow around the hard phases and then re-intersects.

In Figure 8 a detail of an isolated grain can be observed. Cracks at grain boundary split and begin to grow also on grain boundary. The crack aspect, at higher magnification, appears jagged suggesting that it was deflected by grain boundary precipitated phases – in Inconel 718 the most important being the δ phase.

The morphology of δ phase can be film like, when it precipitates at the lower temperature region of the precipitation domain, while at higher temperatures the phase grows along favorable habital planes in the matrix and the film like morphology disappears.

The morphology of δ phase was observed by Sjoberg as film at grain boundary when exposed for 16h at 800°C and left to cool at room temperature and plate morphology when over aged for 62h at 930°C [10]. In our case the temperature was 850°C with stresses, so we would expect an incipient plate like morphology in the material. Depletion of Nb in δ phase vicinity results in a weakened region of material which provides a favorable path for crack extension, as we were able to observe.

The presence of plate like morphology of δ phase – serrated grain boundaries – creates obstacles for crack growth and an improvement of mechanical properties is to be expected. In Figure 9 a serrated grain boundary is depicted, the δ phase appears as a discontinuous film, the morphology tending to become plate [10,12].

The δ phase has a detrimental effect on the yield stress but no effect what so ever on the tensile strength at room temperature and at 550°C [1] and it guides the crack to grow in a zigzag pattern along the grain boundaries on which it precipitates [2,3]. This zigzag pattern, not that obvious, was also present at studied cracks, a good detail of this aspect can be observed in Figure 8. The area surrounding the δ phase is more

Figure 1: Macroscopic aspects when polished for hardness tests.

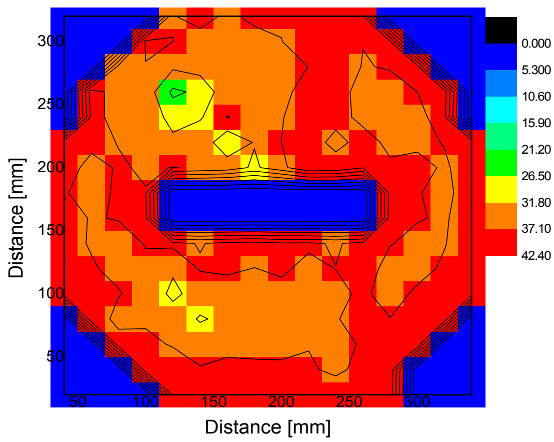

Figure 2: HRC map on the die surface.

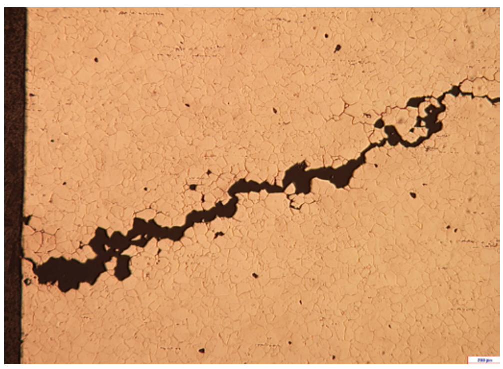

Figure 3: Intergranular crack in Inconel 718 in the aperture corner.

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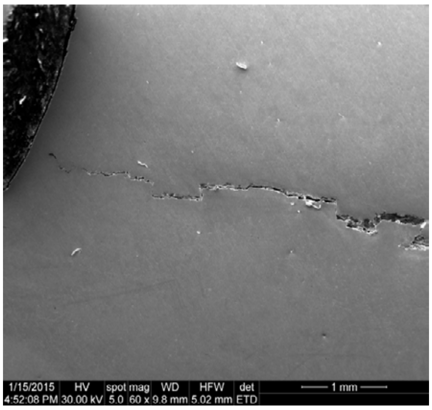

Figure 4: SEM micrograph of the main crack.

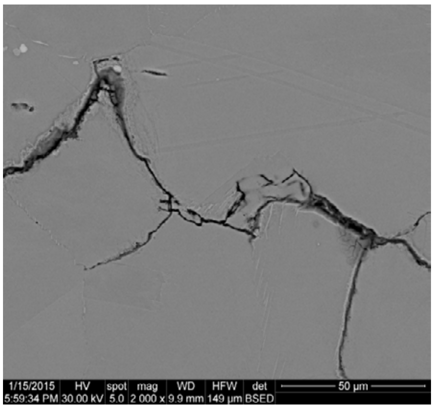

Figure 5: SEM micrograph with details of the main crack.

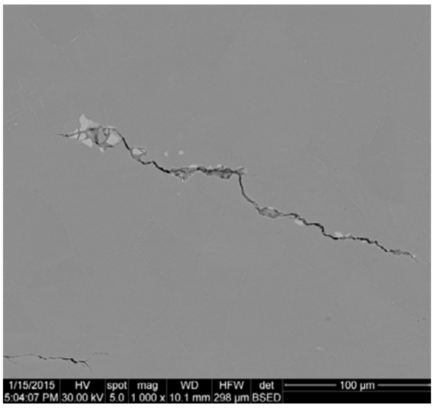

Figure 6: SEM micrograph showing a crack with no exterior contact

plastic than the reinforced matrix – the region is depleted of reinforcing phases [13-15].

When an elemental map was performed, as presented in Figure 10, titanium and niobium carbides were found embedded in an alloyed austenitic matrix. Also, a fine detail regarding small spacing between the carbides was found, the path on which the crack grew.

When the median region of the die was investigated the alloy microstructure was found to be normal, with polyhedral twinned austenitic grains. Strings of inclusions were found (Figure 11) aligned and parallel to working direction of the material.

High temperature oxidation in air enhances damage effects, local changes in chemical composition and microstructure occur, a high sensitivity to high temperature intergranular oxidation in terms of intergranular crack initiation and propagation can be expected from Inconel 718 [15,16].

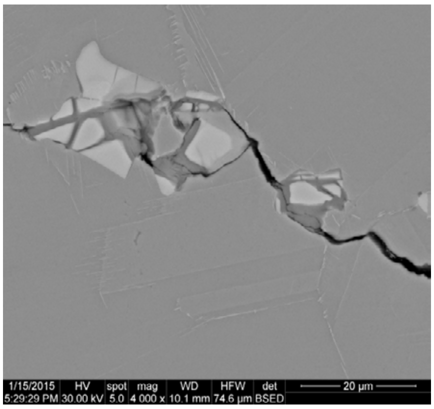

Figure 7: SEM micrograph showing crack interactions with phases present in Inconel 718.

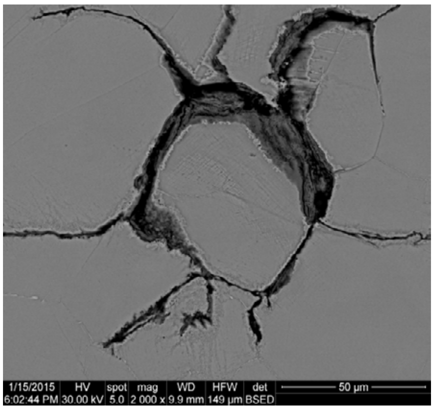

Figure 8: SEM micrograph of an isolated grain.

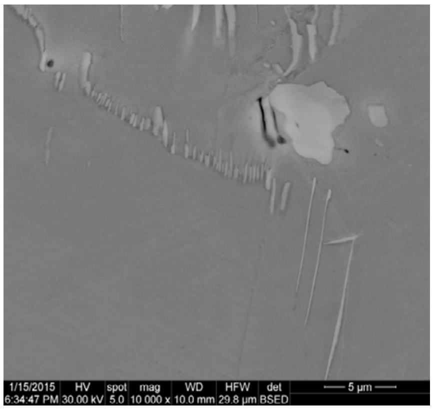

Figure 9: SEM micrograph showing grain boundary.

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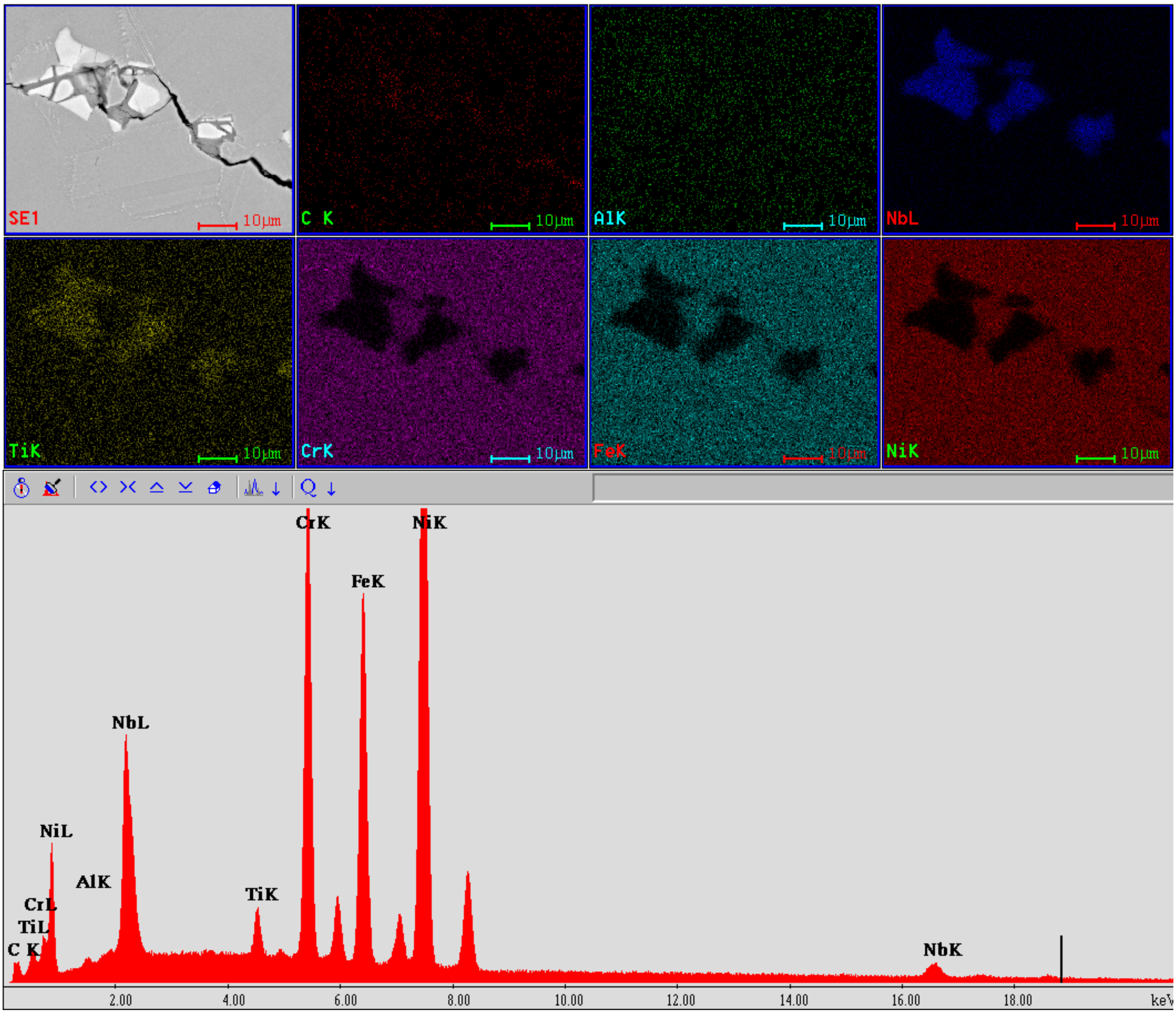

Figure 10: Elemental mapping and EDAX spectra at the interaction between crack and phases.

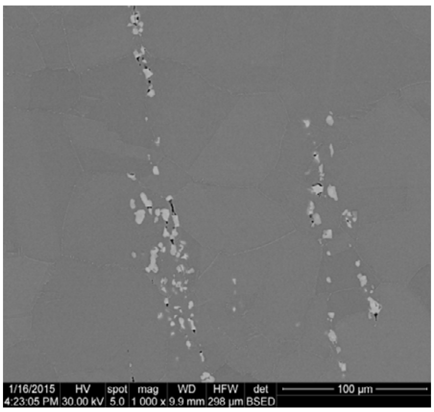

Figure 11: SEM micrograph on a sample from the median region of the die

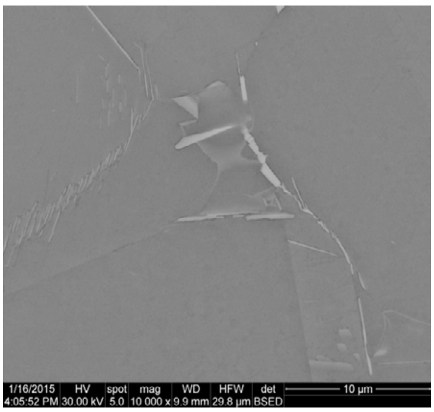

Figure 12: SEM micrograph of precipitated phases a grain boundary.

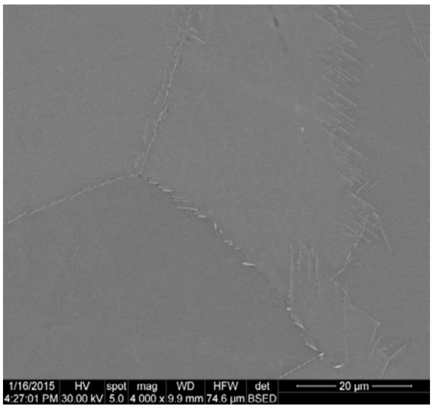

Figure 13: SEM micrograph detailing serrated grain boundaries.

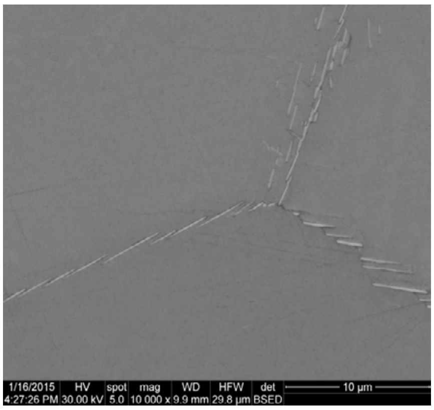

Figure 14: SEM micrograph showing plate or needle like phase present at grain boundary.

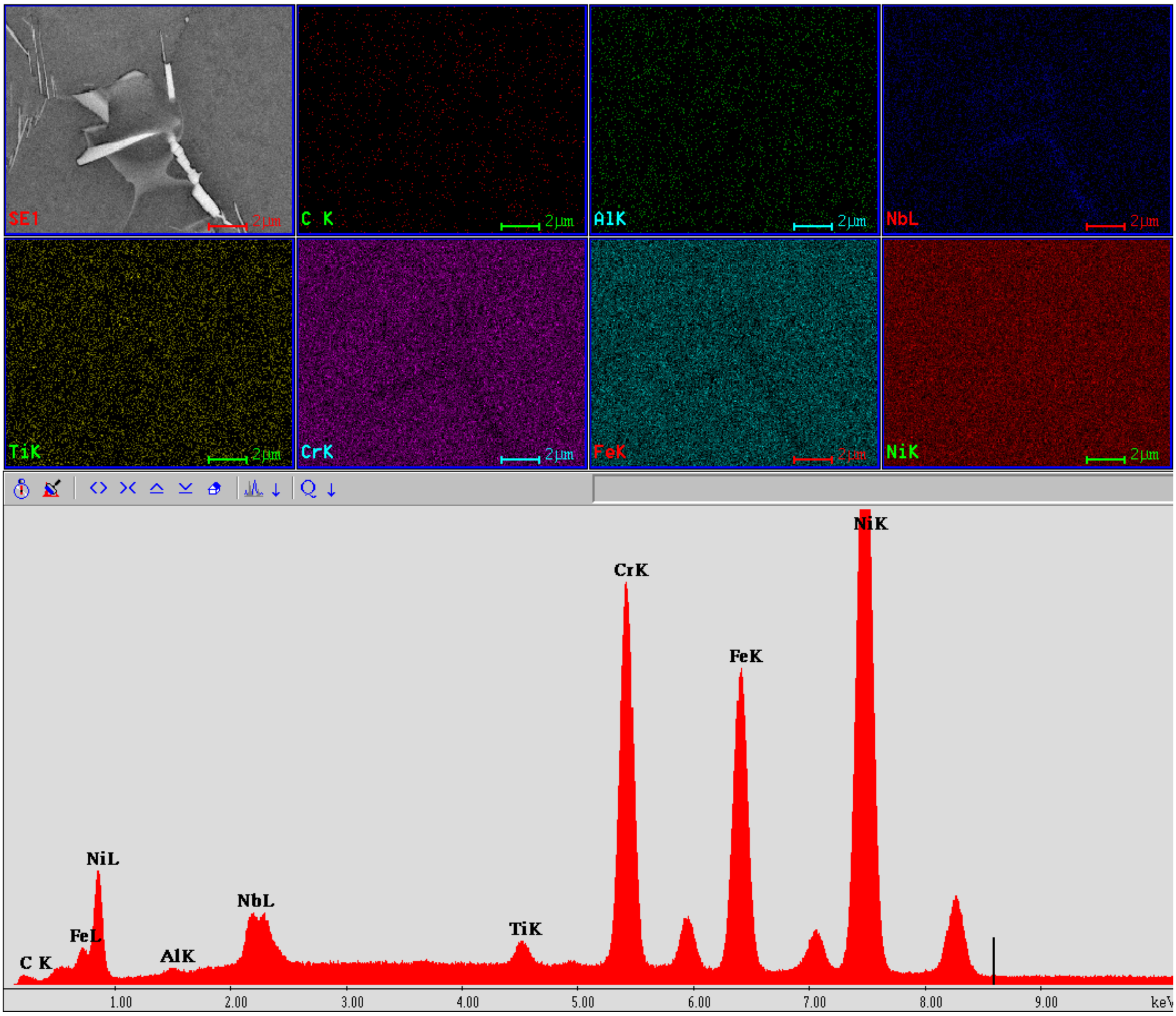

Figure 15: Elemental mapping and EDAX spectra on grain boundary.

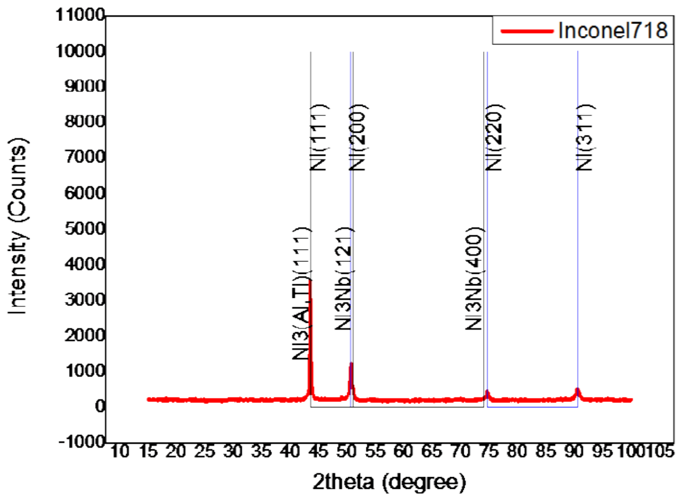

Figure 16: XRD pattern of Inconel 718.

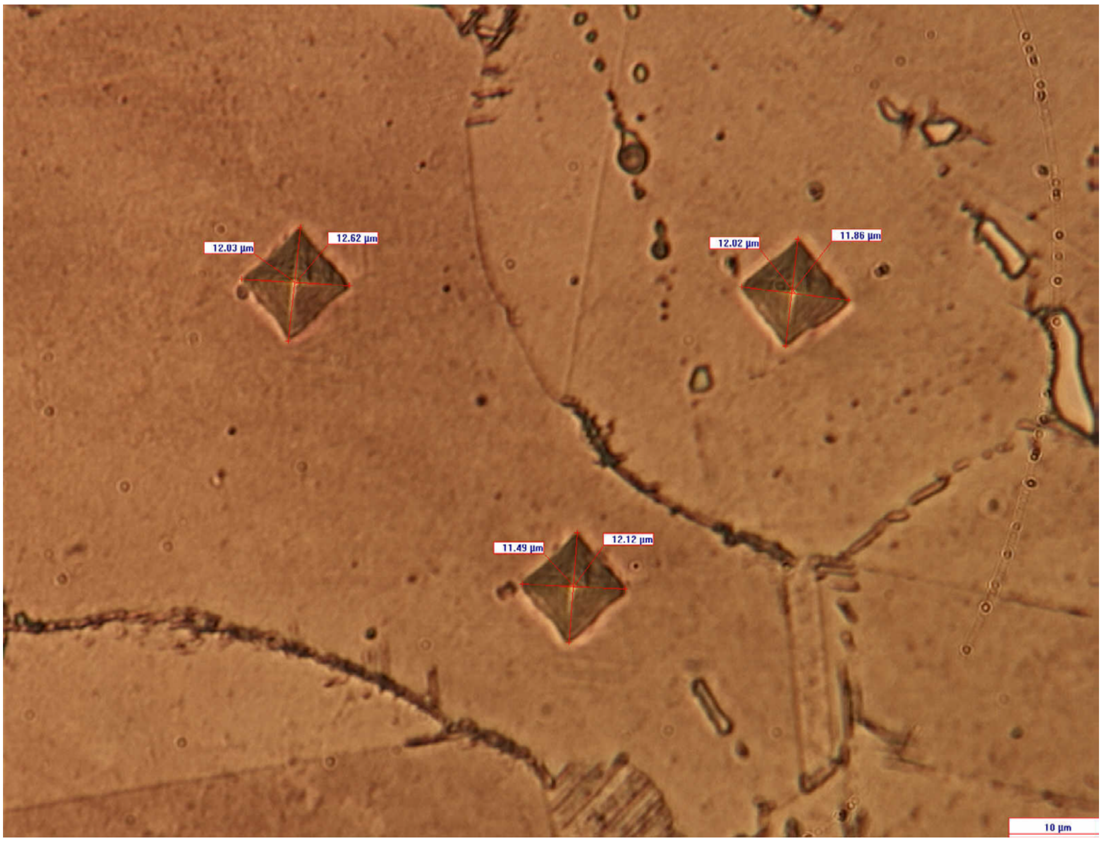

Figure 17: Microhardness imprints in the matrix.

Interaction between oxygen and crack tip is considered by two theories, the stress accelerated grain boundary oxidation in which grain boundary oxidize in front of the crack tip and crack opening creates new surfaces for oxygen exposure and the dynamic embrittlement which supports the idea of grain boundary embrittlement by oxygen diffusion [17,18].

These mechanisms may explain cracking phenomena when the crack starts from die aperture and grows towards the middle of the die, while smaller hairline cracks which occur deeper and have no exposure to air have different morphologies and may propagate in the depleted matrix.

At grain boundary, especially at higher magnification (Figures 12-14) the presence of a phase which gives the serrated aspect was found. As previously observed, based upon morphology and location, this phase was assumed to be the δ phase.

As the δ phase content increases the high temperature plasticity of Inconel 718 decreases [1]. For Inconel 718 the main hardening precipitate is γ'' with same chemical composition as δ (Ni_3Nb). Near precipitated δ a region with low volume fraction of γ'' would appear and this zone will have lower mechanical characteristics.

The δ phase precipitated at grain boundary increases crack growth and high temperature stress rupture characteristics of the alloy [10]. Still, the present stage, based upon δ phase morphology and size, the alloy finds itself in an acceptable condition for use.

In the elemental map from Figure 15 an alloyed austenitic nickel matrix can be distinguished and grain boundary phases, due to their size, cannot be detected. Still, a small region could be captured in which a high niobium and nickel concentration was present, while other alloying elements were absent. This aspect strengthened our assumption that this phase would be the Ni_3Nb δ phase.

A sample was tested by X-Ray diffraction, the resulting XRD – pattern is presented in Figure 16. Mainly strong peaks from the nickel austenite phase were observed similar aspects presented [18]. On closer inspection of the pattern multiple phase presence could be inferred, but because the resolution was not acceptable we were unable to determine precise phase concentration.

This analysis revealed the possibility of $Ni_3(Al,Ti)$ gamma prime and Ni_3Nb δ phase peaks but no phase quantification was possible. The matrix was indexed as a FCC phase with a lattice parameter of 3.610Å. Dehmas [19] reports a value of 3.604Å. A phase separation procedure is required for accurate phase quantity determination [20].

The major strengthening phase is gamma double prime and even a small volume fraction, lower than 25%, is enough for the alloy to be a high – strength alloy. The volume fraction of gamma prime is usually less than 5% and its contribution to strengthening the alloy is incidental [21-25]. Also peak shifts were observed which lead to the conclusion of strains present in the material. These strains also could not be accurately determined.

The microhardness tests performed on the samples, an example presented in Figure 17, used a Vickers indenter and a 40 g load. The mean Vickers microhardness was at 513 with a 23 standard deviation. All tests were performed on the matrix.

Conclusions

We found that a die made of Inconel 718 would be more effective in hot copper extrusion. The extruded quantity was 40t, 8 times higher than when usual steel dies were used. The main reason for die discarding would be aperture decalibration by plastic deformation and cracks which in this current stage did not affect die performance.

Further use of this die could have been made by enlarging the die aperture and producing a larger product – the microstructure in median region of the die would have allowed further use. If we were to assume a same amount of extruded material, i.e. 40t with an enlarged aperture, the Inconel 718 die would allow us to extrude 16 times more copper.

By optic and mainly scanning electron microscopy the cracks were observed. They appeared intergranular, typical for high temperature cracking, with a jagged aspect due to δ phase presence at grain boundary. The median region of the die revealed a normal structure for the alloy.

Current die performance is found to be acceptable for production in S.C. LAROMET S.A., the extruded quantity was increased per die. Industrial trials and further researches on Rene 41 made dies are currently performed expecting a better result, since the limit temperature for Inconel 718 is below the copper extrusion temperature used in the factory.

Acknowledgment

These research results are obtained within the project entitled 'Materiale special destinate confectionarii matritelor pentru extrudarea la cald a cuprului si alamelor' (Special materials for hot extrusion dies for copper and brass), an effort from Politehnica University of Bucharest and LAROMET S.A.

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