A Comparative Microstructure Evolution of AISI H21 and Inconel 718 in Cyclic Heating

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The AISI H21 steel used for dies in copper hot extrusion softened only after several runs. To improve production parameters a new material was necessary for the die, and, as a first choice, Inconel 718 was preferred. Prior to adopting this material for new extrusion dies an experimental program with temperature exposure cycles was performed so as to study microstructure evolution within AISI H21 and Inconel 718. The samples were studied by optic and scanning electron microscopy (SEM) and were also submitted to X-ray diffraction (XRD) and microhardness tests. The results showed carbide coalescence, which led to softening, followed by a re-precipitation in the steel, while, in Inconel 718, the precipitation (in small amounts) of delta (ä) phase was observed.Using these results and combining them with practical experience with steel, we could predict that the Inconel 718 die performance is such that at least a double quantity of copper could be extruded per die.

Keywords: AISI H21, Inconel 718, extrusion die, δphase, carbide coalescence

The most employed hot work tool steels – forging tools, extrusion dies, die casting moulds - are the AISI H grades [1]. Unfortunately, the temperature limit for these steels is around 600°C since above 500°C microstructural changes occur. For copper hot extrusion, in our case, the billet temperature is around 850°C and significant changes in tool steel microstructure occur. These changes cause steel die failure by thermal fatigue, wear, softening and creep [2].

Previous research allowed us to identify the steel die failure mechanism for our particular application: copper hot extrusion die softening caused by carbide coalescence and matrix depletion of reinforcing elements [3].

Methods for an increase in the die life span were studied, such as a careful monitoring of the die hardness, to identify the moment when the microstructure was severely altered and restore the right one by heat treatment. This simple solution could not be implemented since the production line had to be stopped in order to change the die, perform the heat treatment and all the subsequent operations. It was found to be more convenient to expand the die opening and use it for a larger extruded product. Another method considered was die cooling but, given the current equipment, the die wall thickness would have been severely reduced and thus it would no longer have been fit for the stresses encountered during extrusion.

The technological limitations and economic aspects were overcome by considering a new alloy for die manufacture: Inconel 718, a nickel-based precipitation-strengthened superalloy.

The service temperatures for this alloy - up to 923K (approximately 650°C) [4] - are higher than those of steel, yet bellow the temperatures used for copper hot extrusion (850°C in our case).

Designed to be used at temperatures higher than 540°C, the microstructure for Inconel 718 is comprised of an austenitic continuous matrix (γ), reinforcing phases, gamma prime (γ'), gamma double prime (γ''), carbides, borides and other topologically close packed phases. In nickel-iron superalloys, the gamma double prime, an ordered phase rich in niobium, is the strengthening phase.

In Inconel 718, the ä phase (Ni_3Nb), with an orthorombic structure and acicular shape, appears when overaged in the temperature range 815-980°C [5].

Researches performed by Yuan et al. [6] showed that by aging at 900°C for 1-24h, in the microstructure of Inconel 718, along γ, γ' and γ'', the δ phase appears, precipitated during this treatment.

As temperature exposure time increases, the percentage of δ phase increases and the percentage of γ'' (the reinforcing phase) decreases and, as consequence, the decrease of the peak stress and strain is to be noted. The δ phase has a softening effect [6].

The δ phase precipitates first at grain boundaries, then on twin boundaries and last within the grain. Cai et al. [7] considers the optimal shape for the δ phase as rod-like and small. Long needles, when present within microstructure, decrease the alloy mechanical properties.

The mechanical characteristics are optimal up to 550°C, followed by a drop, and above 760°C are no longer adequate for extrusion purposes. The characteristics of the steel used were acceptable up to 350°C, followed by a severe drop as temperature increases.

With this in mind, our main concern was when and if δ phase forms in copper hot extrusion temperature regime and, if choosing Inconel 718 as a material for extrusion dies, it would increase the production.

The experimental program was devised so that only the temperature effect on the microstructure was studied. It must be assumed that stresses present during extrusion will accelerate ä phase precipitation at twin boundary and within the grain. The alloy will soften faster.

Experimental part

The chemical composition of the alloys is given in table 1 and 2, as specified by the producer.

Cubic samples with similar volumes and sizes were cut using a metallographic cutter -five samples for each alloy.

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Table 1
AISI H21 CHEMICAL COMPOSITION

%C	%Si	%Mn	%Cr	%Ni	%Mo	%Cu	%V	%W	%Fe
0.300	0.309	0.312	2.26	0.235	0.132	0.119	0.282	7.53	Bal.

Table 2
INCONEL 718 CHEMICAL COMPOSITION

%C	%Cr	%Mo	%Ni	%Ti	%Al	%Nb	%Fe	%Si	%Mn
0.022	17.73	2.96	Bal.	1.00	0.49	5.18	18.43	0.06	0.06

A heating simulation was performed to validate the experiment and establish heating cycle parameters. The procedure is presented as follows.

The experiment consisted of inserting the samples (at room temperature) in a preheated furnace at 850°C and placing them on a 30mm thick steel plate kept always within the furnace. The samples are kept for 300 s in contact with the 850°C preheated steel plate, extracted and placed on an insulating material and left to cool until room temperatures. The same facet of the cube was used to contact the steel plate and the insulating material during the whole experiment.

These operations are repeated for 100 heating cycles. At each 20 cycles a sample was removed and analyzed. Visual inspection did not reveal any cracks or distortions of the samples- only a thin, non adherent oxide layer formed on the surface which was removed with a piece of cloth after each cycle.

Prior to the experimental program a dilatometric analysis was performed to identify critical points for the material.

The metallographic specimen preparation for the experimental samples followed the typical steps; the etchants used are Nital 5% for steel and glyceregia (15mL HCl+10mL glycerol+5mL nitric acid) for Inconel 718.

First observations were performed on an optical microscope, followed by SEM observations. The samples were also investigated by XRD and, finally, microhardness tests were performed.

Results and discussions

For AISI H21 steel the expansion curve analysis revealed the transformation temperature at 796°C and 831°C with no particular aspect- the curve was presented in [3].

For Inconel 718 the expansion curve is depicted by figure 1.

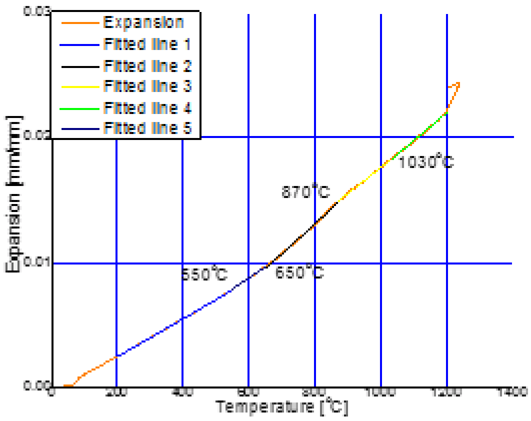

Fig. 1 Expansion curve for Inconel 718

The curve was differentiated and points on the curve were found where most likely a transformation was to occur. On each temperature interval a line was fitted to find the slope to confirm a shift.

The first shift of the expansion curve was found at 550°C. This shift can be attributed to an eventual precipitation of reinforcing phases. There are different opinions in literature, some authors [8] consider that at temperatures between 550 and 660°C γ' and γ'' precipitate at large aging times while other authors consider higher temperatures 700 to 900°C with shorter exposure times [9, 10]. There is still uncertainty about the phase transformations in Inconel 718, several authors [9 - 14] reporting different temperatures and aging times. At early stages the precipitates are too small to be identified by conventional techniques. The slope change at 550°C is considered by Slama [12] to be caused by precipitation of γ'.

A slight increase in expansion is also found around 650°C. Inspecting the time-temperature-precipitation (TTP) diagram reveals that around this temperature the γ'' precipitates. Another shift of the expansion curve- indicating a contraction, was found around 870°C, which can be associated with the appearance of the δ phase on the grain boundary. Finally, a further contraction can be identified on the expansion curve around 1030°C and it can be attributed to the dissolution of the δ phase. Similar research and results can be found in [14, 15].

The next step involved sample preparation for metallographic analysis. Conventional methods for each alloy were employed to reveal the microstructure. On unetched specimens the inclusions were observed: both alloys showed a low content in inclusions which were aligned and parallel to working direction.

Microstructure changes are observed in AISI H21 samples beyond a 40 cycle exposure. When compared with initial microstructure few or no changes could be observed.

The structure appears martensitic, with fine carbides dispersed within this matrix, as shown in figure 2. At 60 cycles a lower bainitic microstructure should appear, and carbides should be larger in size (fig. 3), aspects also mentioned by Nurbansari et al.[16]. The carbide coalescence occurred.

Fig. 2 AISI H21 initial structure, nital etch, martensite with fine carbides

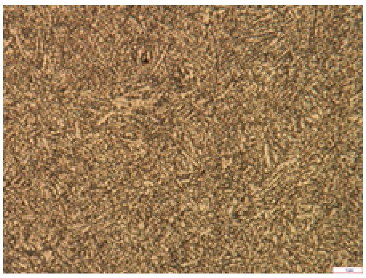

Fig. 3 AISI H21 structure after 60 cycles, nital etch, lower bainite and carbides

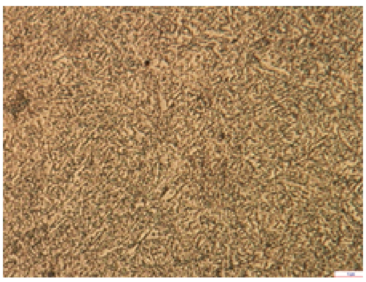

Fig. 4 AISI H21 structure after 80 cycles, nital etch, lower bainite and coalesced carbides

After 80 cycles the proportion of bainite is considered to increase and the microstructure change can be seen in figure 4. At 100 cycles the structure suffers obvious changes. Heating the alloy above 700°C is a process similar to a fourth tempering. Alloy carbides form along with a hardening of the steel and the re-crystallization of martensite is complete. Equiaxed grains form and tend to grow extensively, as seen in figure 5.

Fig. 5 AISI H21 structure after 100 cycles, nital etch, equiaxed grains and large carbides

According to changes in microstructure a heat treatment applied to the die when its microstructure would resemble that observed at 40 cycles would increase the lifespan of the die. In practice a careful monitoring of the hardness would clearly show the right time to perform the heat treatment, but in the current factory working regime this would not be applicable- the production would be stopped.

For Inconel 718 the structure appears austenitic (γ nickel) with twinned grains and aligned MC carbides. Also Ti(C, N) can be observed in figures 6 - 9 the grain boundary appears serrated due to the presence of probably low proportions of the δ phase and carbides.

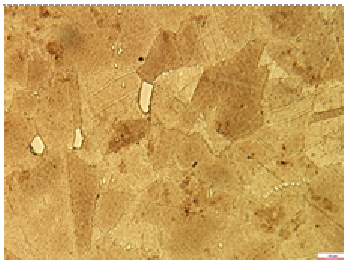

Fig. 6 Inconel 718 as received, glyceregia etch, the austenitic matrix with titanium carbides

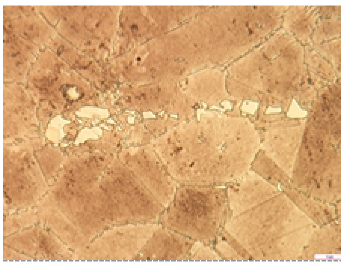

Fig. 7 Inconel 718 the sample exposed for 60 cycles, glyceregia etch, austenitic matrix and aligned carbides

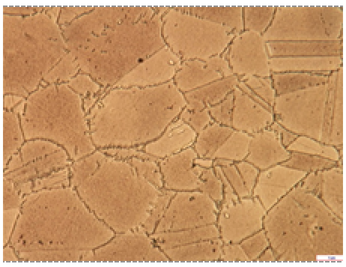

Fig. 8 Inconel 718 the sample exposed for 80 cycles, glyceregia etch, austenitic matrix and serrated grain boundary - precipitated delta phase

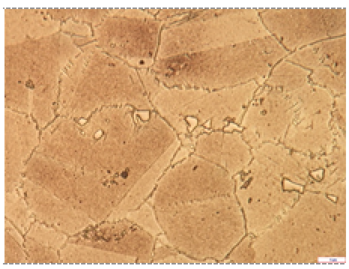

Fig. 9 Inconel 718 the sample exposed for 100 cycles, glyceregia etch, austenitic matrix and serrated grain boundary.

By optic microscopy no obvious changes in microstructure were observable. If compared with API standards [17], the microstructures appear to be acceptable throughout all heating cycles.

A grain size measurement according to ASTM E-112 96 was performed on Inconel 718. Grain size variation in connection to the number of exposures is presented in figure 10 and no variation was observed.

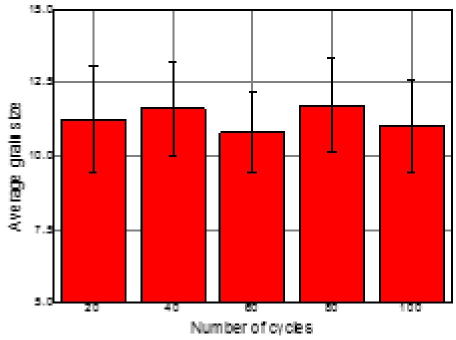

Fig. 10 Average grain size variation in connection to the number of cycles. Error bars represent one standard deviation

In SEM studies it was observed that in AISI H21 the martensitic matrix does not appear very clear and the dominance of a high fraction of carbides finely dispersed within the matrix can be observed in figure 11-13. The M_6C carbide appears rod-like and spherical in shape, aspects similar can be observed in the studies of Nurbansari et al. [16]. The W content in the M_6C increased since the carbides grew and became stable (fig. 14).

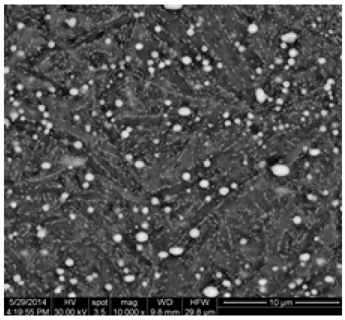

Fig. 11 SEM micrograph on AISI H21, the sample exposed for 20 cycles

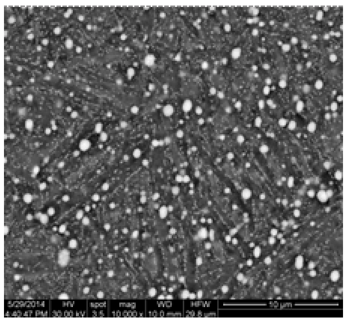

Fig. 12 SEM micrograph on AISI H21, the sample exposed for 60 cycles

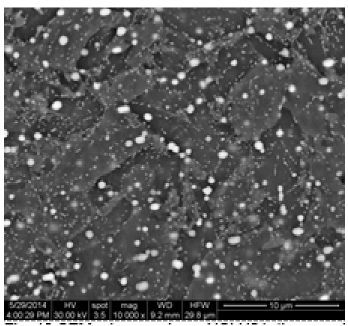

Fig. 13 SEM micrograph on AISI H21, the sample exposed for 100 cycles

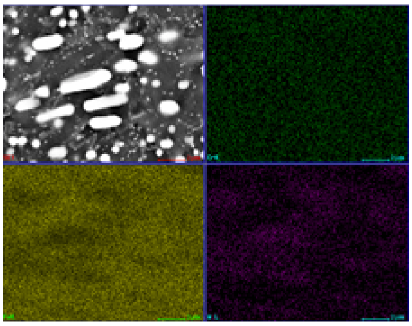

Fig. 14 Elemental map performed on carbides

When tempering above 600°C the number of carbides decreases since they coalesce and re-crystallization and growth of grains occurs and the morphological feature of martensite vanishes. The shape of martensite, when tempering, is maintained up to 650°C. To be noted is that mainly carbides on former austenitic grain boundaries grew by coalescence.

Carbides prevent grain boundary sliding and migration by pinning and strengthening the boundaries. When the matrix was depleted of fine carbides the die would have failed by softening and plastic deformation-the microstructure of the analyzed failed dies appeared similar to the microstructure of the 60 cycles exposed sample.

Further temperature exposure (for 80 and 100 cycles) shows a secondary carbide precipitation. These carbides appear nanometric in size and aligned on former martensitic needle boundaries.

By close monitoring of the die hardness a favorable stage to perform a heat treatment could be identified and the die life could be increased.

In Inconel 718 the metal matrix is austenitic (γ nickel) with twins and carbides in the metal matrix, as seen in fig. 16.

The elemental map shown in figure 15 revealed (Nb, Ti) carbides and titanium nitrides within the austenitic matrix. Grain boundary δ phase (rod and needle shaped) precipitates are clearly observable at 80 cycles (fig. 17).

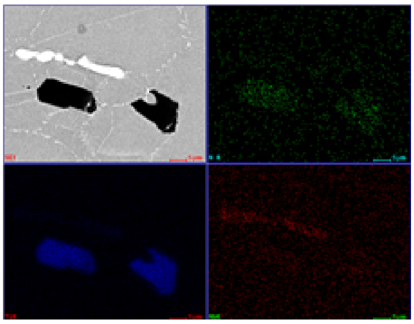

Fig. 15 Elemental map on carbides

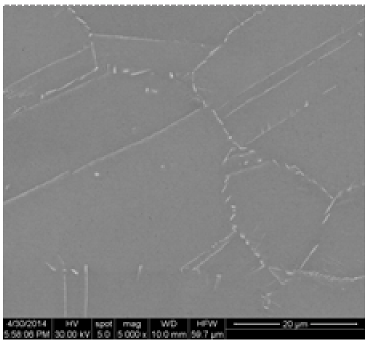

Fig. 16 SEM micrograph on Inconel 718, the sample exposed for 20 cycles

Also, a platelet δ phase was observed at grain boundary at higher magnifications, similar aspects being presented by Kuo et al. [18]. The amount of the δ phase increased slightly as the total temperature exposure time increased as can be observed when comparing figure 17 and figure 18.

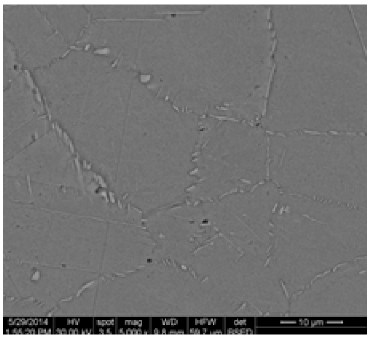

Fig. 17 SEM micrograph on Inconel 718, the sample exposed for 80 cycles

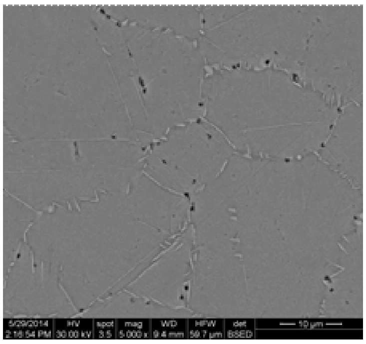

Fig. 18 SEM micrograph on Inconel 718, the sample exposed for 100 cycles

On grain boundaries precipitation of chromium carbides ($Cr_{23}C_6$) can be inferred, but further investigations are necessary.

By XRD on AISI H21 (the diffraction patterns shown in fig. 19) one observed that the carbides were predominantly of M_6C type, tungsten rich with a face centered cubic structure, and the presence of several MC type carbides [19] could also be inferred. After 100 cycles a peak appeared at 84° which could be associated with the $M_{23}C_6$ carbide.

Nurbansari et al. [19] suggested following carbide evolution: Matrix $\rightarrow Fe_3C \rightarrow M_6C$ and an in situ transformation which occurs simultaneously Matrix $\rightarrow Fe_3C \rightarrow M_{23}C_6$.

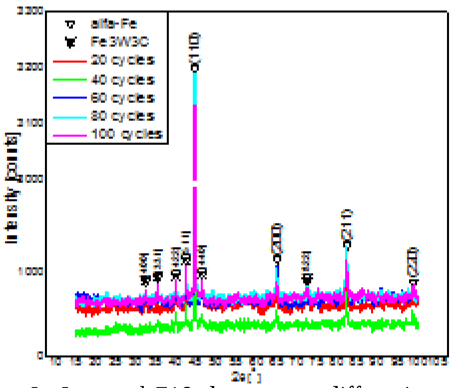

Fig. 19 Superimposed XRD patterns of all samples from AISI H21

In Inconel 718 the strong diffraction peaks of γ phase with a face centered cubic crystal overlap with the $Ni_3(AlTi)$ over the whole range of 2θ degrees, the XRD pattern is shown in figure 20.

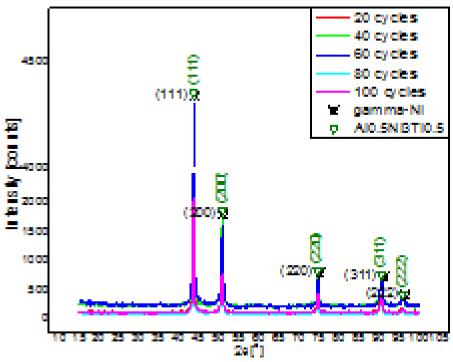

Fig. 20 Superimposed XRD patterns of all samples from Inconel 718

The XRD diffraction pattern performed on extractions from the precipitates presented by Chang et al. [20] confirm the overlapping with the matrix specific peaks. Their presence causes a broadening of the peak at the base due to coherency strains. In this case, when the Rietveld method was employed to determine the phase content it failed to converge and further studies were performed assuming the existence of a single phase, i.e. austenite (γ nickel).

By this assumption a lattice parameter was determined and its variation with the number of heating cycles depicted in figure 21.

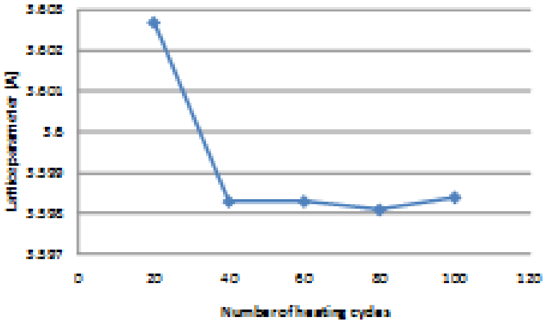

Fig. 21 Lattice parameter variation for Inconel 718

Lattice parameters determined by Cozar [9] for the matrix (γ) were found at 3.601±0.001A for Inconel 718 (1200°C/1h/water quench) and 3.594±0.002A (aging at 750°C/524h/water quench). The determined values from the experimental XRD pattern are consistent with those determined by [9], even with the low accuracy introduced by the assumption. The lattice parameter dimension dropped after 40 heating cycles and remained at steady values. This decrease suggests a structure relaxation caused by a thermal effect similar to a heat treatment or by diffusion of some elements.

The microhardness tests for steel show a drop after 60 cycles (fig. 22). The microstructures corresponding to the hardness may be either pearlitic either pearlite-baintic, as suggested by Mutlu [21]. The higher hardness suggests a martensitic structure, followed by a bainitic structure. The pearlite was not observed. The increase in hardness after 60 cycles could be attributed to a precipitation of secondary carbides which was observed in the SEM studies.

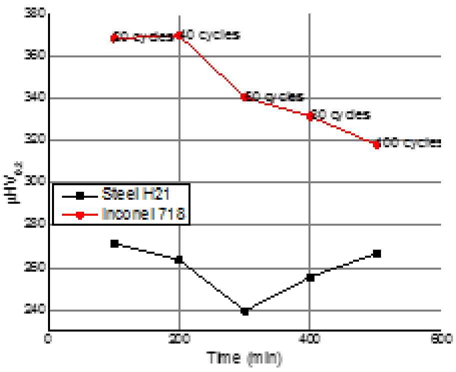

Fig. 22 Microhardness variations on tested samples (time - number of cycles equivalence is shown).

Inconel 718 shows descending microhardness values. When aged at 953K for different durations of time El-Bagoury's research [22] revealed an increase in hardness related with longer exposure times – 335HV30 for 150 hours aging time. This increase can be explained by the precipitation of the γ' and γ'' phases.

The decrease in hardness can be explained by the precipitation of the δ phase, which normally would occur at an exposure to 850°C beyond 10 h. Precipitation was observed, in our case, only after a total time of 2 h exposure. The processing history of the alloy was not made available, so this find was useful.

Precipitating δ platelets deplete the reinforcing δ'' phase around them, and if NbC should precipitate at grain boundaries the region adjacent to them would be also depleted in γ'' by the diffusion of Nb and the bonding with carbon.

Conclusions

This research was performed in order to establish if choosing Inconel 718 over AISI H21 as a material for extrusion dies would improve significantly the production per die.

Using dilatometric analysis critical temperatures for Inconel 718 were identified - helpful to find new phases appearing in microstructure. Metal carbides and nitrides and the δ phase appeared on SEM images; neither the reinforcing γ prime nor γ second could be identified by XRD. The peaks of reinforcing phases overlapped with the peaks of the austenitic matrix – their presence was inferred by broadening the peak base. No grain size change was observed in Inconel 718.

For steel a single peak appeared after 100 cycles which could be attributed to $M_{23}C_6$; all peaks were found for M_6C or the matrix.

It was established that:

-After a 20-cycle exposure a slight microhardness drop was observed for AISI H21, but no obvious changes in microstructure occurred. In Inconel 718 no changes occurred except for a decrease in the lattice parameter which should suggest a relaxation, but, bearing in mind the considerations made, it cannot be stated as certain.

-After a 40-cycle exposure steel microharndess continues to drop, whereas in Inconel no changes occur. No obvious microstructure changes occur in both materials.

-After 60 cycles the steel microhardness drops to its minimum value- the microstructure changes, lower bainite appears, carbides grow and deplete the matrix of the reinforcing phase. Inconel 718 shows also a hardness drop and the γ phase precipitates at the grain and twin boundary; still the microstructure appears acceptable according to the API Standard [23].

-After 80 cycles the steel shows an increase in hardness -secondary carbide precipitation occurs. Carbides, nanometric in size, appear aligned on former martensitic needles reinforcing the matrix. Inconel 718 continues to show a decreasing hardness and an increase in the δ phase content.

-After 100 cycles the steel continues to increase in hardness to its second hardness peak in this study. Carbides continue to precipitate in the metal matrix. Inconel continues to show decreasing microhardness values, the δ phase content increases, yet the microstructure is acceptable according to standards.

Practical experience using a steel die allows a mean quantity of 4000kg is extruded/die which means 25 runs. The total time in which the die contacts the 850°C billet equals 2.08h.

In our experiment the die would fail after 60 cycles - 5 hours exposure at 850°C, when carbides grow by coalescence and deplete the matrix of reinforcing elements. In practice, when the stresses from extrusion are present, the time would fall below half.

For Inconel 718 the microstructure showed promising results, thus a die was produced and put into service. Until now 40.000kg of copper have been extruded, totaling 250 runs and 20.8 total exposure times at 850°C and no signs of softening occurred. Only slight cracks propagating from the corners of the die can be observed.

A cost analysis for the two dies was performed. The steel die costs 799 euro with an average production of 5tons (with die repairs included), while the Inonel 718 die costs 845 euro and it withstood for 40tons of copper extrusion; since this is the first die used, we will consider this to be the average. Dividing the die costs to the production, steel dies would yield 160euro/ton while the Inconel 718 die would yield 22.37euro/ton.

In the current stage of the research, using Inconel 718 as extrusion die material proved to be an efficient choice in economic terms.

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