IJE TRANSACTIONS C: Aspects Vol. 32, No. 6, (June 2019) 852-859

International Journal of Engineering

Journal Homepage: www.ije.ir

Transmission Electron Microscopy Sample Preparation of INCONEL 738 Nickel-Base Superalloy

F. A. Hesari*, M. Moradi, F. S. Torknik

Materials Characterization Group, Materials and Energy Research Center, Meshkin Dasht, Karaj,. Iran

P A P E R I N F O

Paper history:
Received 26 December 2018
Received in revised form 03 February 2019
Accepted 07 March 2019

Keywords:
INCONEL Alloy
Jet Electro-polishing
Ion Beam Milling
Micro Control Dimpling

A B S T R A C T

Size, shape, volume fraction and distribution of embedded γ' phase in γ phase has direct effect on strength of INCONEL alloy. Microstructure parameters of INCONEL phases are quantified from microstructure images using transmission electron microscopy (TEM). Different TEM sample preparation techniques were used to study INCONEL 738 alloy microstructure for transmission electron microscopy (TEM). The INCONEL 738 was first cut into a 1 × 1 cm slice with 600 µm thickness with diamond wire cut. INCONEL sample was mounted by wax (M135), after initial grinding and polishing. The molded INCONEL sample was further polished by different grit size SiC paper to reduce the thickness below 80 micron. At this stage, 3 mm discs were cut from the thin slice of INCONEL alloy by mechanical punch machine. The 3 mm discs of INCONEL alloy were used for TEM sample preparation. Three methods of electro-jet polishing, ion milling and micro control dimpling were employed to prepare transparent TEM sample to observe the surface microstructure details of INCONEL 738 alloy. Electro-jet polishing TEM sample preparation technique could reveal microstructure details of INCONEL alloy γ and γ' phases using 42% H_3PO_4, 34% H_2SO_4, 24% H_2O electrolyte at about – 40^0C bath temperature and applied voltage of 30 V.

***doi**: 10.5829/ije.2019.32.06c.08*

1. INTRODUCTION

Complex nickel base superalloys are widely used in different application requiring strength at high temperature and fracture toughness. Rotating blades in gas turbine is the example of such an application in high temperature critical materials. The high temperature strength of INCONEL alloys is attributed to the γ' [Ni_3(Ti, Al)] phase in the matrix [1]. This phase is precipitated in a continuous matrix of nickel with face centered cubic (FCC) structure (γ phase). γ' Phase [Ni_3(Ti, Al)] is appeared as a fine dispersion of quasi-spherical particles. The mechanical properties of Ni base superalloys are strongly depended upon the morphology, size and distribution of γ' precipitate in Nickel base matrix [2]. The γ' precipitate coarsening can occur during initial heat treatment and also in subsequent service duration. A small misfit or defects in crystallographic structure of γ' and γ phases can cause massive microstructural change at elevated temperature. Therefore, it is important to evaluate and predict γ' [Ni_3 (Ti, Al)] and γ (Ni base) phases coarsening and particle dissolution kinetics in order to improve high heat resistance of such alloys [3]. For this, different theoretical models have been developed to predict coarsening kinetics of γ' [Ni_3 (Ti, Al)] phase [4]. In all these models, average γ' precipitate radius are measured from SEM and TEM microstructure images with increasing aging time during coarsening and compare with model calculated average γ' precipitate radius prediction. SEM and TEM studies are used to observe the primary and secondary phases (γ and γ') and determine the size, volume fraction, inter-particle spacing and distribution of phases in the matrix [5, 6]. Model prediction of γ' phase coarsening kinetics can be evaluated by SEM and TEM microstructure image quantification [7].

The electron microscope is an essential tool in materials science research for obtaining microstructure details or topography of materials. Electron microscope

*Corresponding Author Email: *f-a-hesari@merc.ac.ir* (F. A. Hesari)

Please cite this article as: F. A. Hesari, M. Moradi, F. S. Torknik, Transmission Electron Microscopy Sample Preparation of INCONEL 738 Nickel-Base Superalloy, International Journal of Engineering (IJE), IJE TRANSACTIONS C: Aspects Vol. 32, No. 6, (June 2019) 852-859

can be generally divided in two groups of transmission and scanning electron microscopes. Microstructural observation can be done by transmission electron microscope where majority of electrons beam should be able to pass through thin sample without much distortion. Scanning electron microscope is used to obtain topography image of a surface. Although two types of microscopes have similar principal in electron source emission, focusing, diverging and converging of electron beam but image formation mechanism is quite different in transmission and scanning electron microscopes. In transmission electron microscope, image contrast is formed by transmitted electrons through sample. Scanning electron microscope image is formed by emission of backscatter and secondary electrons from sample surface.

Sample preparation technique is an important step in having good image in electron microscope beside setup technical abilities. Image contrast is very much depended on sample preparation techniques in transmission electron microscope since electrons transmit through thin sample structure. Therefore, sample preparation techniques are the most important and crucial step to obtain true information from material microstructure. INCONEL series microstructure has been investigated by SEM and TEM since development of nickel based superalloys [8].

In SEM sample preparation, samples are ground to 800-1000 grit and mechanically polished or electropolished and then etch with etchant solutions. For metallic elements and alloys, etchant solutions are predominately acid or peroxide-containing solutions. Solutions like HCl- HNO_3 and H_2O or CrO_3- H_2SO_4 and H_3PO_4 are used as etchant solutions. Etching is either done electrolitically or by immersion of sample in the etching solution [9]. For TEM observation, samples are ground to 60- 80 μm and then electro polish in electrolyte solution. Different electrolyte solutions were reported in literature for INCONEL alloys series for TEM and SEM sample preparation; such as 10% $HClO_4$, 25% C_2H_5OH and 65% C_4H_9 OH [10]; 10% $HClO_4$, 20% glycerol in methanol for INCONEL 713C [11]; 10% perchloric acid, 90% ethylic alcohol for INCONEL 718 [12]; 10%$HClO_4$- 90%C_2H_5OH -7g thiourea in 100 ml solution [13], 35.6 ml H_2SO_4-37.5 ml HNO_3 and 9.4 ml H_3PO_4 [14]. In SEM investigation of INCONEL 738LC, sample was polished electrolytically in a solution of 45% butanol, 45% acetic acid and 10% perchloric acid at 20-25 V for about 25 s and then immediately electroetched in the same solution at 4-5 V for a few seconds [15]. Beside TEM sample preparation equipment's performance, hands-on experience is also an important parameter to have appropriate thin TEM sample. TEM image quality is improved on electron transparent sample with least surface artifact [16]. There are various type of preparative techniques which can be chosen based on material's type and intended structural information. A general rule of thumb is that the higher the atomic number elements in sample, scatters electron more. These samples are needed to be thinner than samples have lighter atomic number elements. All sample preparation techniques aimed towards creating a foil type area with a thickness of about 10 nm. Thinning of TEM sample may be subdivided into the three steps i) Initial thinning to make slice of material between 100 to 200 μm by metallographic techniques ii) cut 3 mm disk from the thinned slice of sample iii) thinning central region from one or both faces of disk. Three major thinning methods are jet electro-polishing, ion beam milling and precision microprocessor control dimpling. Most of the electrically conducted metal samples are thinned by electro-polishing method.

Ma [17] investigated effect of different TEM sample preparation techniques of INCONEL alloy 783 microstructure observation. He concluded that jet electro-polishing can produce a large artifact-free electron transparent region, wherein INCONEL alloy microstructure details can be well observed. He also pointed out that ion beam milling technique causes amorphous structure and surface damage to sample. Therefore, ion beam milling TEM sample preparation can be considered as a rough method for INCONEL alloy surface microstructure study. Focused ion beam technique have been developed to overcome extensive surface artifact develop by traditional bulk ion beam milling technique [18]. However, focused ion beam milling has its own limitation.

TEM and SEM investigations of INCONEL alloys were reported in the literature but no article was sited in literature to study INCONEL 738 TEM sample preparation in a systematic procedure. In this work, INCONEL 738 TEM samples were prepared using three techniques, jet electro-polishing, ion beam milling and precision dimpling. The effect of different TEM sample preparation techniques on the TEM microstructure imaging were compared and evaluated.

2. EXPERIMENTAL MATERIALS AND PROCEDURE

INCONEL 738 is a heat resistance alloy with composite structural material. The chemical composition of INCONEL 738 alloy is complex one and mainly consist of C, Cr, Co, Nb, Ti, Ta, Al, Mo, Fe, Mn, Sr, Zr balanced Ni. Elements like sulfur, silicon are also exist in chemical composition of INCONEL 738 in very minor amounts. Table 1 shows percent composition of INCONEL 738 alloy.

High heat resistant property of INCONEL 738 alloy is directly related to stability, phase composition and structure of alloy. The main phases of INCONEL 738 alloy are intermetallic γ' phase ($L1_2$ type) based on

TABLE 1. Chemical composition of INCONEL 738

Elements	Percentage (%)
C	0.15-0.20
Cr	15.7-16.3
Co	8.0-9.0
Nb	0.6-1.1
Ti	3.2-3.7
Al	3.2-3.7
Mo	1.5-2.0
Fe	0.5 max.
B	0.005-0.015
Ta	1.5-2.0
Mn	0.2 max.
Ni	rest

Ni_3Al and nickel solid solution (γ phase, FCC type). The % elemental composition of γ′ and γ phases are differed from each other.

In nickel solid solution phase, elements like cobalt, chromium, molybdenum and titanium act as a strengthening agent. Chromium is also act as an oxidation protective agent in alloy. Long term strength of INCONEL 738 alloy is improved with increase in γ′ phase volume fraction and its distribution in γ phase which is proportional to % content of aluminum in alloy composition. MC type carbide phase is also formed in INCONEL 738 alloy with that containing carbon like NbC, TiC which complemented γ phase in composition.

MC type carbide phase is initialized formation of secondary $M_{23}C_6$ type carbide with help of γ′ phase during operation at high temperature. Formation of close pack intermetallic phases is undesirable phenomenon in INCONEL alloy structure since it decreases stress rapture and strength of alloy. Therefore, interaction of several factors are involved to develop a heat resistance structure in INCONEL alloy. Morphology, dispersion and volume fraction of γ′ phase morphology and dispersion of γ phase, MC and $M_{23}C_6$ type carbides are among important factors that should be observed from surface topography.

The INCONEL specimen was cut in size of 10.0 mm×10.0 mm by wire cut machine (Spark). The thickness of sample was about 600 μm at this stage. The surface damage depth was reduced by using diamond wire cut tools. Speed of wire saw, abrasive size and feed rate have to be selected appropriately for less surface damage.

The specimen was then mounted with wax (mwax 135) at 90 ^{0}C. At this stage, the surface of specimen was grounded and polished to get a flat face with uniform analysis condition across the region of interest. The wax mold made easy to hold the specimen by hand during grinding and polishing. The specimen edges were also protected in wax mold during grinding and polishing. The next step was to grind the specimen surface on abrasive SiC paper with different gritting size starting from course grit to finer grit sizes. Starting with 50 grit SiC paper, the 80, 120, 400, 1000 grit size SiC papers were used to reduce the thickness of INCONEL 738 alloy specimen. Each SiC grit size produces damages to extend of twice the size of SiC grain. In each step, a finer SiC grain was used to reduce the surface roughness produced in pervious step. This was followed by finer polish, using Al_2O_3 paste with the grain size of 0.05 and 0.025 μm. After final grinding step, no scratches were visible on the surface with naked eyes. The same polishing steps were repeated for other side of specimen.

At this stage, the thickness of specimen was between 50 to 120 μm. A self supporting 3 mm disc was cut by disc punch from thin slice of specimen for final thinning step. Three techniques of electro-jet polishing, microprocessor control dimpling and ion beam milling were used to thin the samples for TEM application.

The 3 mm disc of specimen (50-120 μm thick) was electrolytically polished in a twin jet electro-polishing instrument (Fischione 110). Two solutions of (42% H_3PO_4, 34% H_2SO_4, 24% H_2O) and (10% $HClO_4$, 90% C_2H_5OH, thiourea 7g in 100 ml solution) were used as an electrolyte solutions.

A microprocessor control dimpling instrument (SBT, Model 515) was used to thin center of 3 mm disc of specimen until the sample was perforated at the center.

In ion beam milling technique, the 3 mm disc of specimen was fixed on the working table of ion beam milling (BALTEC RES 100) and bombarded with energetic argon ions. Both the guns were tilted at 10° from top of the sample. The voltage was kept at 5.0 kv until the sample perforated and then the voltage was reduced to 2.5 kv and held for 30 min after perforation.

3. DISCUSSION

All samples were examined with transmission electron microscope (CM200 FEG, Philips) operating at 200 KV. Different electrolyte solutions were reported to be used for INCONEL alloy microstructure features revelation where consist of strong acid [10-14]. Among all reported electrolyte solution, 42% H_3PO_4, 34% H_2SO_4, 24% H_2O electrolyte solution was shown to have good etching effect to make phases visible on surface in electro-polishing instruments. Bath temperature and applied voltage were optimized for best thinning condition to reveal microstructure of INCONEL sample surface.

The results are shown in the form of bad and good surface thinning conditions and the results of intermediary conditions are omitted. As shown in Figure 1, the primary phase was only revealed at 22°C bath temperature and applied voltage of 20 V. No clear image of secondary phase γ′ was observed in Figure 1.

Distribution of secondary γ′ phase was not observed in primary γ phase. It is concluded structure of γ′ phase was eliminated in electropolishing step. This could be due to higher electropolishing temperature or applied voltage. Several attempt was made with 42% H_3PO_4, 34% H_2SO_4, 24% H_2O electrolyte solution at different temperature and applied voltage. Figure 2 shows the primary γ and secondary γ′ phases of electropolished sample at about – 40°C bath temperature and applied voltage of 30 V. It was concluded that bath temperature and applied voltage had diect effect on revealing of γ and γ′ phases on image.

In order to check effect of other reported electrolyte solution, a thin foil of sample was also electropolished using electrolyte solution of 10% $HClO_4$, 90% C_2H_5OH, 7g thiourea in 100 ml at about – 40 ^{0}C bath temperature and applied voltage of 28 V. Figure 3 shows the microstructure details of INCONEL 738 sample.

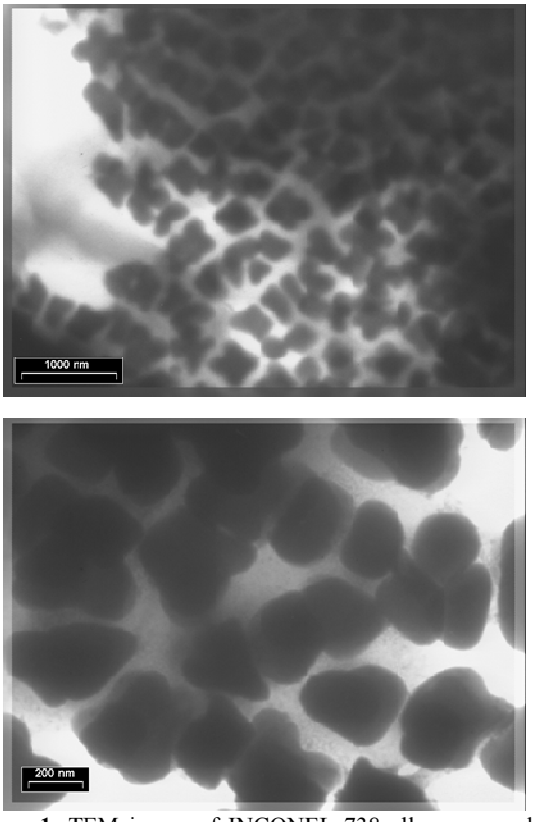

Figure 1. TEM image of INCONEL 738 alloy prepared by Electro-jet polishing using 42% H_3PO_4, 34% H_2SO_4, 24% H_2O electrolyte at 22°C bath temperature and applied voltage of 20 V

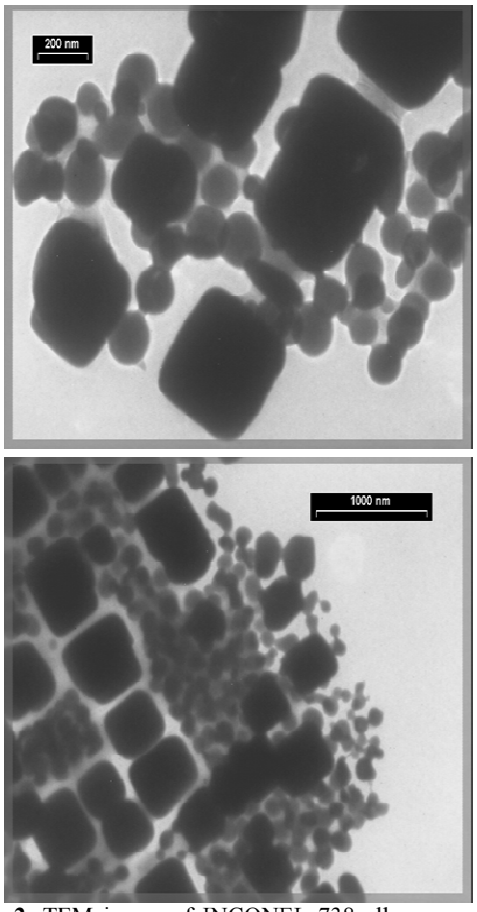

Figure 2. TEM image of INCONEL 738 alloy prepared by Electro-jet polishing using 42% H_3PO_4, 34% H_2SO_4, 24% H_2O electrolyte at about – 40°C bath temperature and applied voltage of 30 V

The microstructure details in Figure 3 is somewhat blurred as compare to revealed microstructure in Figure 2. Detail structure of primary γ and secondary γ′ phases and their distribution are not cleared in Figure 3. It concluded that 10% $HClO_4$, 90% C_2H_5OH, 7g thiourea in 100 ml solution can also be used as an electrolyte solution but parameters like bath temperature, applied voltage and sample thickness have to be optimized with this electrolyte solution again. Primary γ and secondary γ′ phases of INCONEL 738LC were shown with help of high resolution field emission FE-SEM [15]. The distribution of secondary phase is visible in the primary phase in the SEM images.

Figure 2 shows γ′ secondary phase precipitates are well dispersed among γ primary phase. The shape of γ′secondary phase was almost spherical. The shape and morphology of γ′ secondary phase precipitate was much depended to cooling rate of Ni based alloy. It shifted from sphere to cubes, octacubes or octadendrites and dendrites depend on heat treatment condition. Shape transition was depended to cooling rate INCONEL alloy

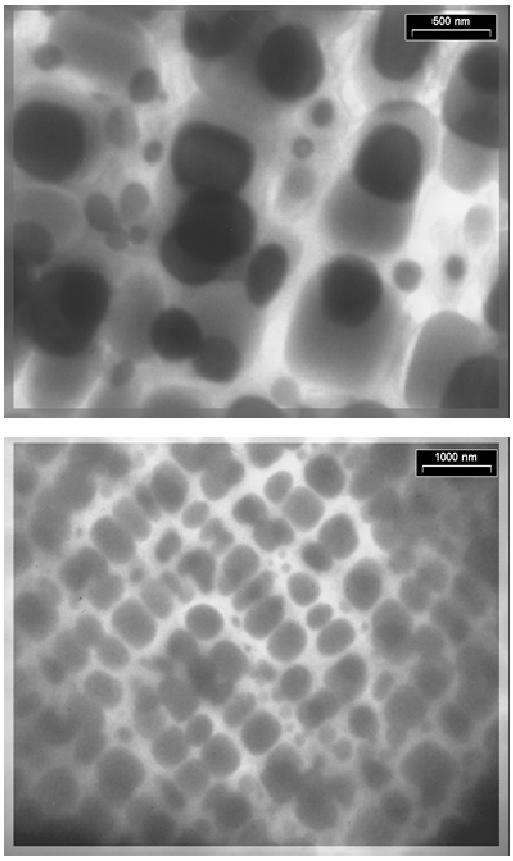

Figure 3. TEM image of sample using 10% $HClO_4$, 90%C_2H_5OH, thiourea 7g electrolyte at about – 40°C bath temperature and applied voltage of 30 V

during heat treatment steps. Clarity of γ′ phase image details among γ phase was quantified through volume fraction, particle size and distribution, shape factor, γchannel width, γ′ and γ lattice misfit parameters. All these parameters had their appropriate effect on INCONEL alloy strength. INCONEL alloy microstructure was controlled by heat treatment procedure (aging temperature and time).

According to Grosdidier [19], spherical shape γ′ phase precipitates was formed among corridors of γ phases at cooling rate of 150 $^oC\ S^{-1}$. γ′ Phase precipitates shape transformation from spherical to cubical was occurred at cooling rate of 100 $^oC\ S^{-1}$. Therefore, shape of γ′ phase precipitates can reveal information about cooling temperature rate. Shape factor of γ′ phase precipitates determined morphological stability of γ′ phase precipitates. It was basically defined as ratio of cross sectional area to perimeter area of γ′ precipitates. Plot of size distribution of γ′ phase precipitates versus their frequency at different ageing time shows effect of aging time temperature on size of γ′ phase precipitates distribution. Shape change of γ′ and γ phases was also induced lattice parameters difference between γ′ and γ phases. This causes a kind misfit between phases which is related to ratio of interfacial energy and coherency stress. Therefore, shape and size of γ′ and γ phases are strongly related to misfits.

Figure 4 shows TEM photographs of the sample prepared by dimpling technique with precise microprocessor control. Thickness of sample was reduced by metallography techniques before dimpling, as explained above. In dimpling instrument, a region was thinned until the sample was perforated near the center. The transparent thin region at perforation edge was used for observation. The transparent thinned area had a gradient toward the center of perforation. The periphery of dimpled thin area was very small as compare to the electropolished thinned sample. The microstructure details of the dimpled sample were not revealed at low magnification as shown in Figure 4. The transparent area was in minimal amount for electron transmission in dimpling process with respect to electropolishing process. Figure 4 shows some kind deformation occurred through perforated area. INCONEL alloy phases had different strength to the mechanical damage and thinning applied to the surface of specimen.

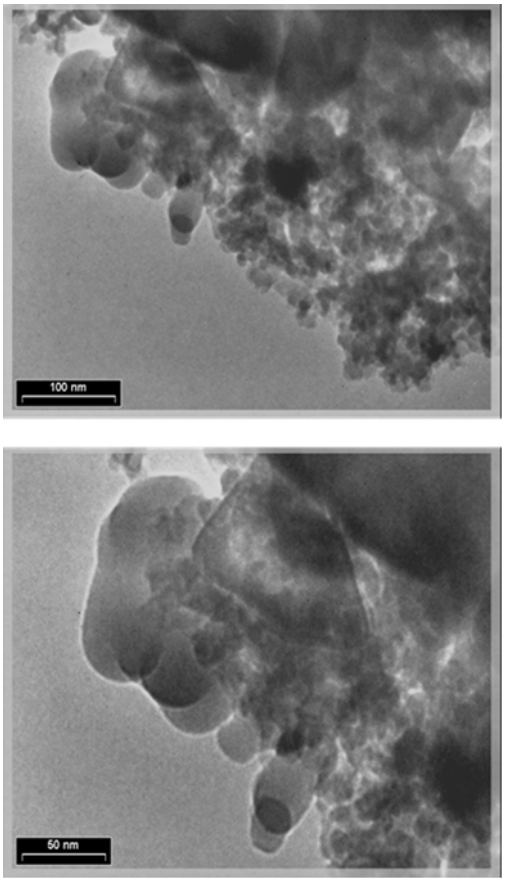

Figure 4. TEM image of sample was precisely dimpled with microprocessor control

As shown in Figure 4, no clear feature of phases is observed even at high magnification since differential thinning was occurred toward sample perforated center. Figure 5 shows the TEM photographs of sample prepared by ion beam milling technique. The morphology of phases is not clear in Figure 5.

It was appeared that the whole sample surface was damaged. Ion beam milling uses the argon ion with the energy of 5.0 kv to bombard the surface of sample. In some point of surface, the argon ions were penetrated deep into the surface. This caused preferential sputtering, specimen heating and radiation on the electron transparent region of sample. Since the hardness and strength of phases in the INCONEL alloy were different from each other therefore phases of alloy sputtered at different rates. As a result, no clear morphology of phases was revealed due to surface damage by argon ions. The sample prepared by ion beam milling technique could not produce a large enough electron transparent region near the periphery of the perforation with the gradient toward the center of perforation. The structure details of ion milled region should be observed at high magnification towards inner center of gradient at the edge of the perforation.

INCONEL 738 TEM specimen preparation method had its own specification regarding to material type,

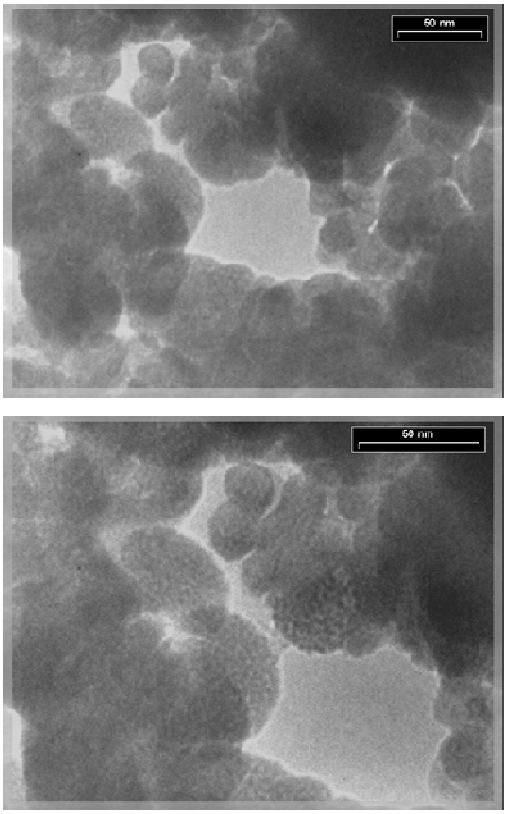

Figure 5. TEM image of sample prepared by ion beam milling

time constrain, equipment availability, skill and information we needed to obtain. In electropolishing, INCONEL specimen was placed in anode and an appropriate voltage was applied to thin INCONEL specimen surface layer by layer. In normal electropolishing thinning practice, electropolishing was proceeded until a perforation (tiny hole) appear on surface of specimen. Surrounding area of perforation was narrow area where surface thickness gradient reduced toward perforation center. In this work, surface perforation of INCONEL specimen were avoided since large electron transparent area of INCONEL specimen would be lost by surface perforation. Patches of electron transparent area were created on INCONEL specimen surface by right adjusting electropolishing parameters. Visual examination of electron transparent area of INCONEL specimen was done by passing light through specimen surface. Using 42% H_3PO_4, 34% H_2SO_4, 24% H_2O as electrolyte solution, a viscous thin film of H_3PO_4 was formed on INCONEL specimen surface to oxidize surface asperities. H_2SO_4 acid was used to dissolve oxides film from surface into a solvent. Film thickness was less above surface protrusions than its valleys. Therefore, surface protrusions was dissolved more rapidly than surface valleys by applying appropriate voltage and electrolyte bath temperature. Potential voltage was set such that no pitting occurred in surface. Cooling of electrolyte was done to slow down thinning process in order not to pit surface. H_3PO_4 film viscosity was also improved in low temperature to avoid a sudden surface pitting.

4. CONCLUSION

Nickel-based alloy INCONEL 738 had a complex alloy with multiplex strengthening mechanisms and corrosion resistance related to γ' and γ phases and carbides. Elemental composition, shape, form and structure of γ' and γ phases and carbides had their contribution on mechanical properties of INCONEL 738. In order to have clear image of γ', γ phases and other precipitates. Microstructure details of INCONEL 738 could be revealed from its thin film (with thickness below 50 nm) by TEM imaging. TEM image contrast of INCONEL 738 was based on mass thickness mechanism in thin film. Incoherent elastic scatter electron (Rutherford elastic scattering) from thin film was cause mass thickness mechanism. Incoherent elastic scatter electron was directly related to atomic number (density or mass) and thickness of thin film. Since film thickness under observation was uniform, therefore image intensity was directly originated from scattered electrons of regions with different atomic number (density or mass). Therefore, γ' and γ phases was easily distinguished from matrix by image contrast.

TEM examination was indicated that the electro-jet polishing thinning technique could produce a large electron transparent area free from artifacts with uniform thickness. Microstructure details of INCONEL 738 was observed with simultaneous presence of γ′ and γ phases. The distribution of γ′ and γ phases could clearly be calculated in a large electron transparent area. Size, morphology of γ′ phase and γ channel width were clearly measurable in TEM image. Exact chemical compositions of γ′ and γ phases can be measured from thin film of INCONEL 738 with help of nanoprobe of electron beam without having matrix effect.

The choice of electrolyte, applied voltage and electrolyte bath temperature had their direct effect on image quality for image information quantification. With precision dimpling technique, a small thin region was obtained at the periphery of perforation with gradient towards the center of perforation. The extent of mechanical damage to the surface was so vast to prevent any micro and nanostructure details observation in TEM. No clear morphology of phases was also observed in a sample prepared by ion beam milling technique due to amorphous damage to the specimen surface. A small transparent area with artifact was obtained in ion beam milling sample preparation technique as compare with electro-jet polishing technique.

This study suggests that the electro-jet polishing technique was the right technique to prepare the TEM sample of INCONEL alloy 738. The proper setting of electrolyte bath temperature, applied voltage and time were also important in getting a large artifact free transparent area with intact microstructure details. Electropolishing is a technology with scientific principles. Beside chemical and process knowledge, it is process of trial and error to handle a specimen.

5. REFERENCES

1. Kulkarni, A., "Additive manufacturing of nickel based superalloy", arXiv preprint arXiv:1805.11664, (2018).
2. Buckingham, R., Argyrakis, C., Hardy, M. and Birosca, S., "The effect of strain distribution on microstructural developments during forging in a newly developed nickel base superalloy", ***Materials Science and Engineering: A***, Vol. 654, (2016), 317-328.
3. Ges, A., Fornaro, O. and Palacio, H., "Characterization of solution and precipitation temperature in cmsx-4 superalloy", ***Procedia Materials Science***, Vol. 8, (2015), 1127-1132.
4. Baldan, A., "Review progress in ostwald ripening theories and their applications to the γ′-precipitates in nickel-base superalloys part ii nickel-base superalloys", ***Journal of Materials Science***, Vol. 37, No. 12, (2002), 2379-2405.
5. Wu, Y., Liu, Y., Li, C., Xia, X., Wu, J. and Li, H., "Coarsening behavior of γ′ precipitates in the γ'+ γ area of a ni3al-based alloy", ***Journal of Alloys and Compounds***, Vol. 771, (2019), 526-533.
6. Malcharcziková, J. and Kursa, M., "Structural characteristics of ni3al based alloys depending on the preparation conditions", ***Metalurgija***, Vol. 54, No. 4, (2015), 635-638.
7. Papadaki, C., Li, W. and Korsunsky, A., "On the dependence of γ′ precipitate size in a nickel-based superalloy on the cooling rate from super-solvus temperature heat treatment", ***Materials***, Vol. 11, No. 9, (2018), 1528-1538.
8. Rogers, B., "Microstructure development in direct metal laser sintered inconel alloy 718", Arizona State University, (2017),
9. Mukhopadhyay, S.M., "Sample preparation for microscopic and spectroscopic characterization of solid surfaces and films", Chemical Analysis-New York-Interscience Then John Wiley-, (2003), 377-412.
10. Zhao, S., Xie, X., Smith, G.D. and Patel, S.J., "Gamma prime coarsening and age-hardening behaviors in a new nickel base superalloy", ***Materials Letters***, Vol. 58, No. 11, (2004), 1784-1787.
11. Zupanič, F., Bončina, T., Križman, A. and Tichelaar, F., "Structure of continuously cast ni-based superalloy inconel 713c", ***Journal of Alloys and Compounds***, Vol. 329, No. 1-2, (2001), 290-297.
12. Slama, C. and Abdellaoui, M., "Structural characterization of the aged inconel 718", ***Journal of Alloys and Compounds***, Vol. 306, No. 1-2, (2000), 277-284.
13. Wanderka, N., Naundorf, V., Banhart, J., Mukherji, D., Genovesse, D.D. and Rösler, J., "Microstructural characterization of inconel 706 alloy", Surface and Interface Analysis: An International Journal devoted to the development and application of techniques for the analysis of surfaces, interfaces and thin films, Vol. 36, No. 5□6, (2004), 546-551.
14. M., G. and Lapin J., "Morphological changes of ϒ' in ni-based superalloy during long term aging", in in 17th International Metallurgical & Materials Conference, METAL 2008, Hradec nad Moravicí, Czech Republic. (2008).
15. Sharghi-Moshtaghin, R. and Asgari, S., "The influence of thermal exposure on the γ′ precipitates characteristics and tensile behavior of superalloy in-738lc", ***Journal of Materials Processing Technology***, Vol. 147, No. 3, (2004), 343-350.
16. Ayache, J., Beaunier, L., Boumendil, J., Ehret, G. and Laub, D., "Sample preparation handbook for transmission electron microscopy: Techniques, Springer Science & Business Media, Vol. 2, (2010).
17. Ma, L., "Comparison of different sample preparation techniques in tem observation of microstructure of inconel alloy 783 subjected to prolonged isothermal exposure", ***Micron***, Vol. 35, No. 4, (2004), 273-279.
18. Li, C., Habler, G., Baldwin, L.C. and Abart, R., "An improved fib sample preparation technique for site-specific plan-view specimens: A new cutting geometry", ***Ultramicroscopy***, Vol. 184, (2018), 310-317.
19. Grosdidier, T., Hazotte, A. and Simon, A., "Precipitation and dissolution processes in γ/γ′ single crystal nickel-based superalloys", ***Materials Science and Engineering: A***, Vol. 256, No. 1-2, (1998), 183-196.

Transmission Electron Microscopy Sample Preparation of INCONEL 738 Nickel-Base Superalloy

F. A. Hesari, M. Moradi, F. S. Torknik

Materials Characterization Group, Materials and Energy Research Center, Meshkin Dasht, Karaj, I. R. Iran

P A P E R I N F O

Paper history:
Received 26 December 2018
Received in revised form 03 February 2019
Accepted 07 March 2019

Keywords:
INCONEL Alloy
Jet Electro-polishing
Ion Beam Milling
Micro Control Dimpling

چکیده

اندازه، شکل، درصد حجمی و توزیع فاز ثانویه γ' در فاز اولیه γ اثر مستقیمی در مقاومت و خواص فیزیکی الیاژ نیکل *INCONEL738* دارد. اندازه‌گیری این دسته از پارامترها برای ارزیابی مقاومت و خواص فیزیکی الیاژ نیکل *738* از رو ی تصاویر میکروساختار با استفاده از میکروسکوپ الکترونی عبوری *(TEM)* صورت میگیرد. سه روش تهیه نمونه الیاژ نیکل *738* برای تصویربرداری در میکروسکوپ الکترونی عبوری استفاده شد. در ابتدا الیاژ نیکل ۷۳۸ به قطعات نازک به ابعاد ۱ در ۱ سانتمتر به ضخامت ٦۰۰ میلیمتر توسط سیم الماسه بریده شد. بعد از عملیات سایش و پولیش اولیه، نمونه در قالب مومی قرار داده شد. در این مرحله با استفاده از ورقه‌های *SiC* با شماره بندیهای مختلف، ضخامت نمونه به ۸۰ میکرون رسانده شد. دیسک‌های به قطر سه میلیمتر از نمونه بوسیله دستگاه پانچ مکانیکی تهیه گردید. از سه روش الکترو جت پالیش، اسیاب یونی و گودبردای در اماده‌سازی نهایی نمونه استفاده شد. در روش اماده‌سازی الکترو جت پالیش با انتخاب محلول٤۲ درصد اسید سولفوریک ، ۳٤ درصد اسید فسفریک و ۲٤ درصد اب در دمای $-40\ ^0C$ و شدت جریان $30\ V$، کلیه جزئیات دو فاز ثانویه γ' و اولیه γ شامل شکل، اندازه و توزیع فاز ثانویه γ' در فاز اولیه γ مشاهده شد. دو روش اماده‌سازی دیگر نتوانست جزئیات کامل میکر ساختار الیاژ را نشان دهد.

***doi**: 10.5829/ije.2019.32.06c.08*