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INFLUENCE OF DIE FORGING PARAMETERS ON THE MICROSTRUCTURE AND PHASE COMPOSITION OF INCONEL 718 ALLOY

WPŁYW WARUNKÓW KUCIA MATRYCOWEGO NA MIKROSTRUKTURĘ ORAZ SKŁAD FAZOWY STOPU INCONEL 718

The object of the present investigation was Inconel 718 alloy. The material in the initial state and after forging at the temperatures of 1100°C and 1000°C was examined. Diffraction analyses indicate that a nickel-based γ solid solution is a dominating phase in the alloy (so-called nickel austenite). Apart from a γ solid solution, which constitutes the matrix, certain volume fractions of the other phase were detected e.g. δ phase and carbides. It was found that, due to thermo-mechanical-treatment at both temperatures, the phase composition of Inconel 718 was considerably changed in comparison to the initial state. On the contrary, differences in the temperature of forging did not significantly influence the alloy constitution. However, both of the temperatures of forging result in distinct texture intensity. Microstructure observations indicate that forging at 1000°C led to recrystallization by creation of the new recrystallizated grains near or on the grain boundaries of existing deformed grains. After forging at 1100°C, the microstructure was fully recrystallized at the whole volume of the material.

Keywords: Inconel 718, microstructure, texture, phase analysis, recrystallization

W pracy przedstawiono wyniki badań wykonanych na stopie niklu Inconel 718. Przebadano materiał w stanie wyjściowym i po kuciu w temperaturach 1100 i 1000°C. Wykonane badania dyfrakcyjne wskazują, że fazą dominującą w stopie jest faza γ – roztwór Fe w Ni, często nazywany austenitem niklowym. Oprócz tej fazy, w stopie występują takie fazy jak δ oraz węgliki. Proces kucia w porównaniu do stanu wyjściowego wpływa na skład fazowy (następuje rozpuszczenie niektórych faz i być może przesycenie osnowy), natomiast różnica w temperaturze przeróbki plastycznej nie wpłynęła znacząco na skład fazowy. Obserwacje mikrostruktury wskazują, że kucie w temperaturze 1100°C prowadzi do pełnej rekrystalizacji dynamicznej, zaś w temperaturze 1000°C rekrystalizacja zachodzi tylko poprzez tworzenie nowych ziarn na granicach ziarn lub w obszarach przy granicach istniejących odkształconych ziarn. Równocześnie, obserwuje się różnice w stopniu steksturowania materiału.

1. Introduction

Nickel-based alloys are modern materials and, due to their characteristics, they are widely used, especially in the aerospace industry. These materials are applied, among others, in the construction of turbine bodies, jet engine components, tanks, combustion chambers, turbine blades and exhaust valves etc. [1-4]. Nickel alloys are used both in casting and plastic deformation technology. These alloys (also known as superalloys) have different chemical compositions [4, 5] and, among them, the one most often used in the industry is Inconel 718 alloy [12].

The most important properties of Inconel alloy are: low thermal conductivity, high hardness and strength at high temperatures and tendency to strengthening [4-6]. The dependence of the mechanical properties of Inconel 718 alloy on the working temperature is shown graphically in Fig. 1. Among commercially available supera-

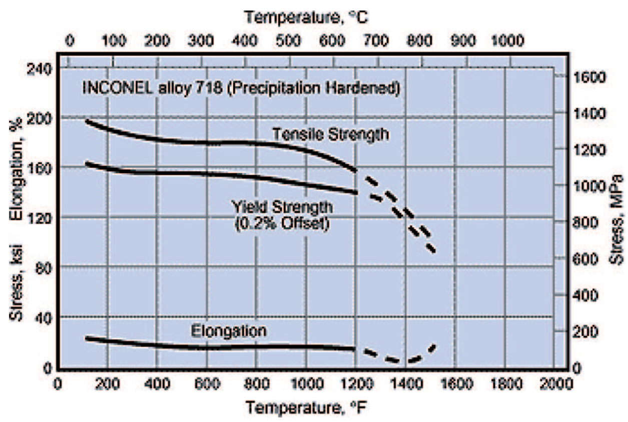

Fig. 1. Change in the mechanical properties of Inconel 718, depending on the working temperature [4]

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lloys, Inconel 718 is one of the most important and most frequently used both in the form of cast (25%) and forged (45%) elements.

The advance in nickel alloys engineering was achieved by adding aluminium to their chemical composition. The addition of this element allows for the creation of intermetallic phases γ' (Ni_3X) coherent with the matrix [7-9]. This phase, having a regular centred cubic structure (A1), strengthens the alloy and makes it stable at high temperatures, what allows for keeping the high strength properties and resistance to creep at high temperatures. The addition of carbon to nickel alloy leads to their strengthening by precipitation through the creation of carbides.

In general, the elements placed at the nickel alloys can be divided into three groups [4]:

- elements involved in the strengthening and stabilization of the alloy – Co, Fe, Cr, Mo, W and V,
- elements involved in the production of the γ' type phases (Ni_3X) – Al, Ti, Nb and Ta,
- elements enhancing grain boundaries – B, Zr, Hf and Mn.

In addition, such elements as Cr, Mo, W, V, Nb, Ti and Ta, form carbides in nickel alloys. Cr and Al also form a compact oxide layer on the alloy surface.

2. Material for the study and research methodology

The subject of the research presented in this study was Inconel 718 alloy having the chemical composition shown in Table 1. As an initial material, rods of a diameter φ =50 mm after casting were used.

TABLE 1

Chemical composition of Inconel 718 alloy

Composition of the alloy [% weight]						
Ni+Co	**Cr**	**Mo**	**Nb+Ta**	**Ti**	**Al**	**Fe**
Investigated Inconel 718 alloy						
53,7	17,9	2,9	5,22	1,0	0,49	Bal.
Inconel 718 composition by the standards [12]						
50-55	17-21	2,8-3,3	4,75-5,5	0,65-1,15	0,2-0,8	Bal.

carbon contents at investigated alloy 0,24%

Round shape samples having a diameter φ = 50 mm and a height H = 30 mm were subjected to forging at the temperatures of 1100°C and 1000°C. These cylinder samples were heated in an electric furnace to the temperature of 1120°C. For forging at 1100°C, the samples were directly transported from the furnace to the forging stage. For forging at 1000°C, the samples were pulled from the furnace and cooled to the desired temperature under controlled conditions (30 seconds) and then placed in the forging die.

Samples for investigations were taken according to the scheme set out in Figures 2 and 3, where the analyzed areas are marked. In this paper, the results of tests for areas 1 and 5 are presented.

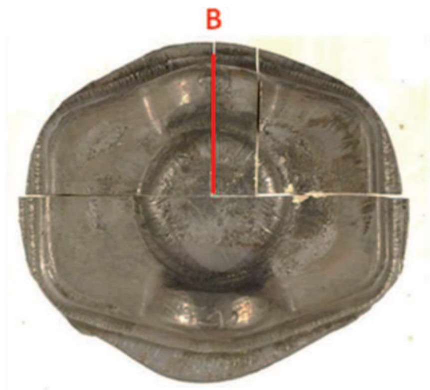

Fig. 2. Macro image of the test sample after die forging (section B)

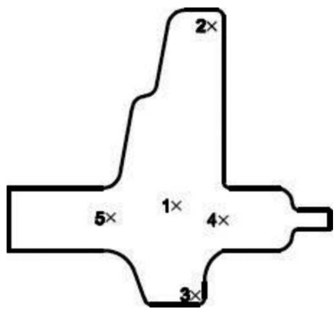

Fig. 3. The cross-section B of die forgings with the areas marked for analysis

The samples were subjected to various x-ray and metallographic studies. Diffraction studies were performed on a Siemens D500 diffractometer, using the monochromatic radiation of a copper x-ray tube ($\lambda_{K\alpha_{śr}}$ = 1.54 Å). Diffraction pattern was determined with the use of Bragg-Brentano method, using step counting ($\Delta 2\theta$ = 0.02°, $\tau_{integration}$ = 6s) in the range of 2θ angle from 30 to 125°. Upon the basis of x-ray measurements for each sample, the qualitative phase analysis and calculation of the texture index were made. Metallographic examination was carried out, using a light microscope Axiovert 200 MAT Zeiss.

3. Results of the investigations and discussion

Phase composition of the investigated alloy

For the samples in the initial state, a diffraction pattern analysis and its comparison with the ICDD database show that the alloy is a material with one dominating γ

phase and additionally δ phase and carbides. The positions of the strongest diffraction lines indicate the presence of one dominant phase in the material, which is a solid solution of iron in nickel (Ni-Fe) –γ phase (Fig. 4). Other phases, whose presence in this material has been found in the initial state, are $NbNi_3$ – δ (orthorombic), and also NbC and TiC (Fig. 4), although the diffraction lines derived from them are very weak.

Heat treatment and forging caused some changes in the phase composition. There is a clear decrease in the amount of the δ phase while the volume fraction of carbides has not been clearly altered (Fig. 5 and 6). The intensities of the peaks coming from intermetallic phases are very small and, therefore, the confirmation or exclusion of their presence in the alloy requires using other methods such as transmission electron microscopy.

In the microstructure of the material in the initial state, equiaxed austenite grains are observed (Fig. 7). Close to the surface of the samples, grains are smaller, indicating a higher degree of the deformation of the surface layer. After forging at the temperature of 1100°C, the material is fully recrystallized by dynamic recrystallization. In the sample area (indicated 1), where the degree of deformation was smaller, grain size is greater than that of the grains in the place where deformation was higher (area indicated 5). After forging at 1000°C, recrystallization is observed only at the area close to the grain boundaries where deformation was higher. (Fig. 8 and 9). Additionally these grater grains at the sample area 1 are equiexed whereas at the samples area indicated 5 are elongated (Fig. 9). Studies using light microscopy reveal the mechanism and extent (degree) of the recrystallization and can also help to explain the observed differences in texture. It can be concluded that both applied to forging temperature led to alloy supersaturation. At the same time, increasing the forging temperature and degree of deformation favours the progress of the dynamic recrystallization. The smaller degree of deformation results in a smaller number of recrystallization nuclei and, consequently, there are larger grains after recrystallization in the structure. The use of a lower forging temperature causes the recrystallization only in the areas near the original grain boundaries (existing in the microstructure before deformation). The new grains, arising from the recrystallization, have not got adequate force to move and grow to consume the interior of the primary deformed grains. Non-recrystallized grains if they remain in the microstructure cause a stronger texture than new grains created at the areas of larger deformation where recrystallization process is more advanced. Similar results were obtained by Yuan and Liu [3], studying Inconel 718 alloy after deformation at different temperatures.

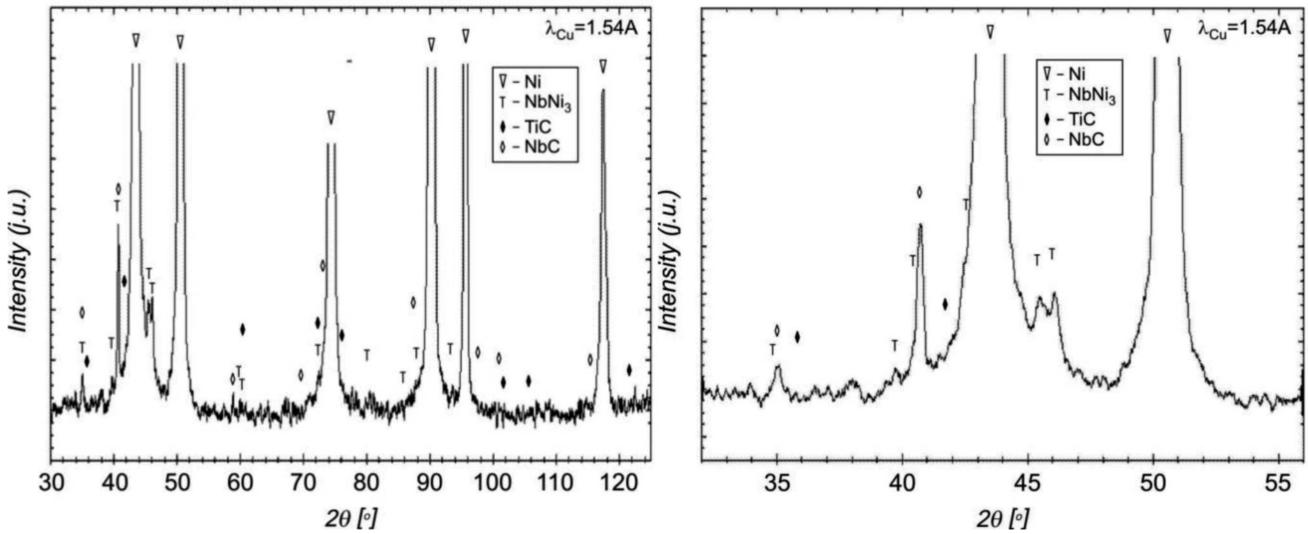

Fig. 4. Diffractogram of a sample from Inconel 718 alloy at the initial state (2Θ range 30-125°, and 30-55° with variable intensity scale to highlight the diffraction lines of phases with lower participation)

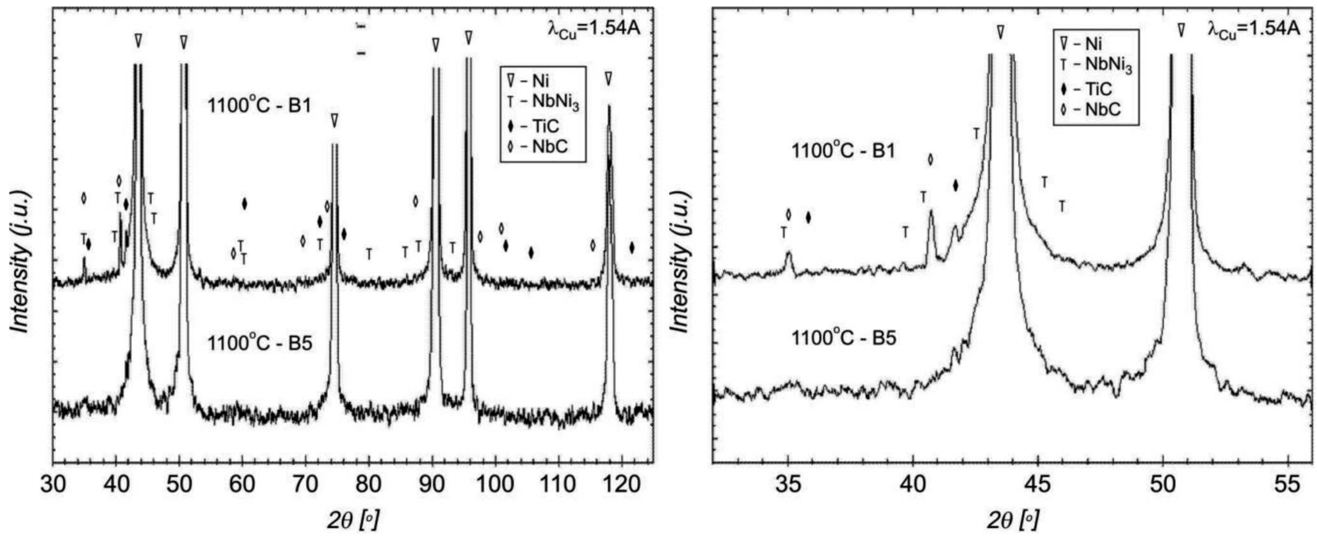

Fig. 5. Diffractogram of a sample from Inconel 718 alloy, after forging at the temperature of 1100°C, in the areas indicated 1 and 5 (2Θ range 30-125°, and 30-55° with variable intensity scale to highlight the diffraction lines of phases with lower participation)

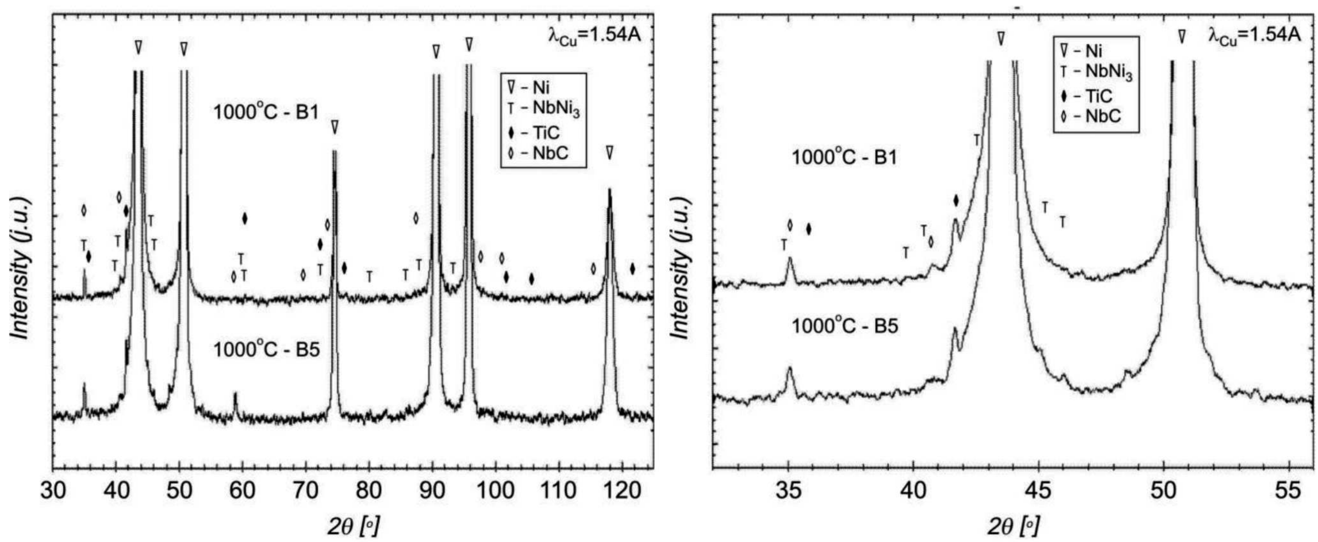

Fig. 6. Diffractogram of a sample from Inconel 718 alloy, after forging at temperature of 1000°C, in the areas indicated 1 and 5 (2Θ range 30-125°, and 30-55° with variable intensity scale to highlight the diffraction lines of phases with lower participation)

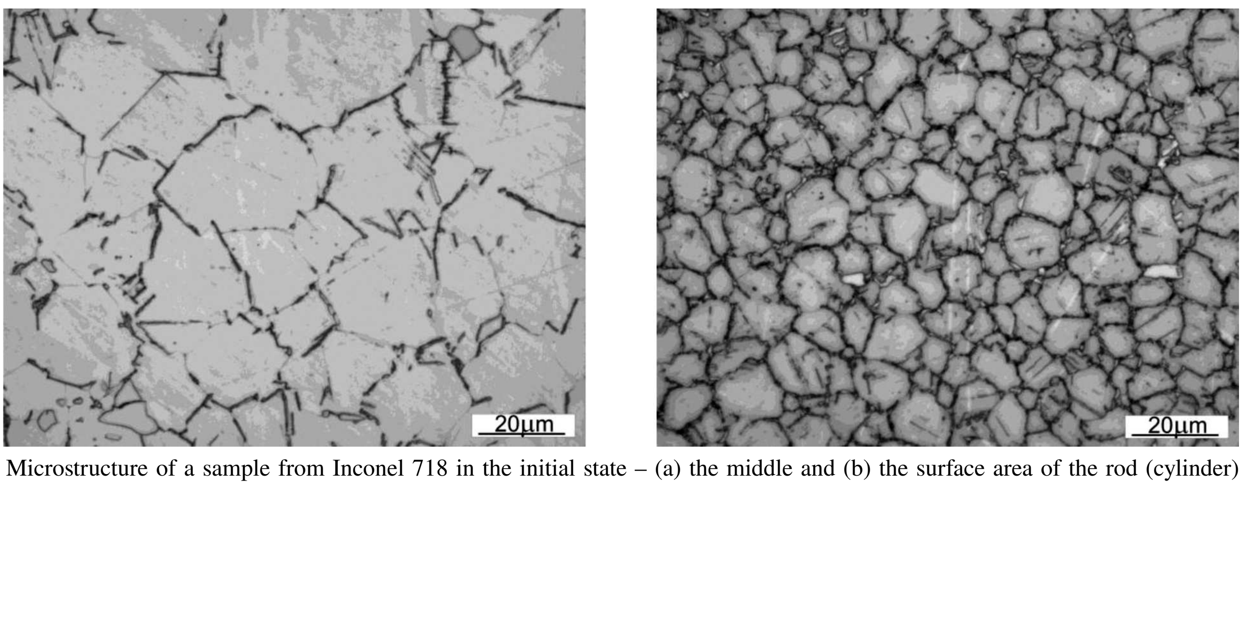

Fig. 7. Microstructure of a sample from Inconel 718 in the initial state – (a) the middle and (b) the surface area of the rod (cylinder)

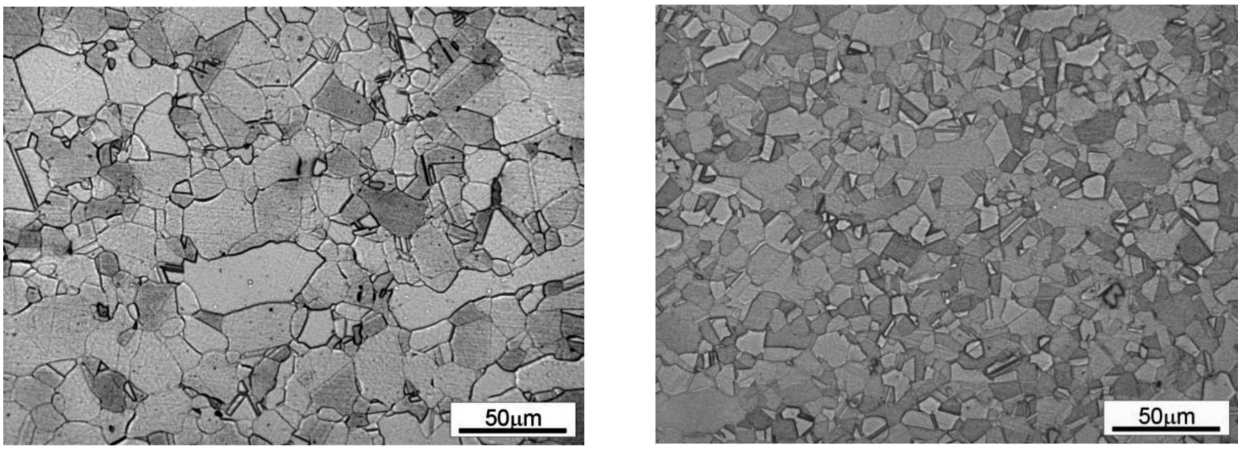

Fig. 8. Microstructure of a sample from Inconel 718 – after die forging at 1100°C, in two different sample areas (a) 1 and (b) 5

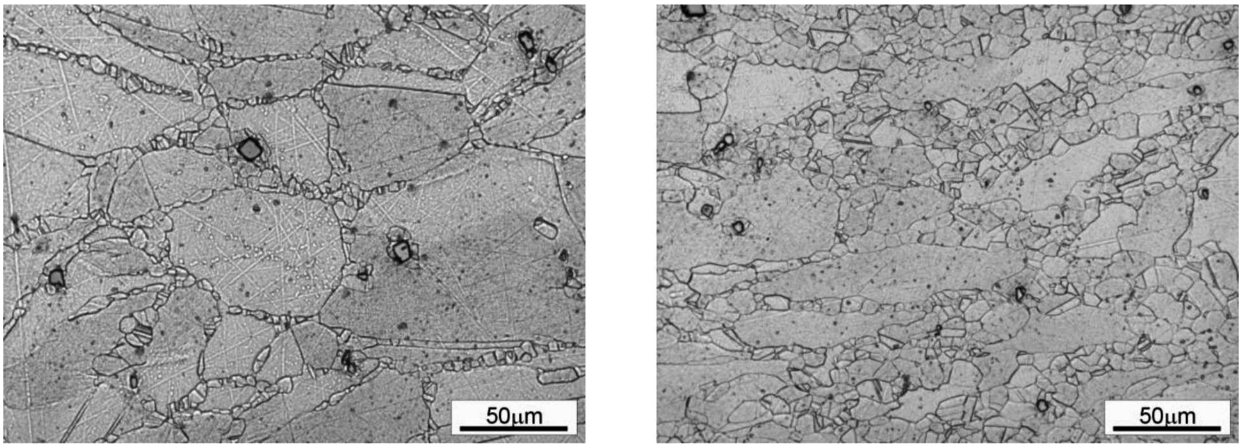

Fig. 9. Microstructure of a sample from Inconel 718 – after die forging at 1000°C, in two different sample areas (a) 1 and (b) 5

The evaluation of the texture degree of the material

The diffraction pattern obtained with the use of Bragg-Brentano method [1,2] allows to determine unambiguously, whether the material is textured, or not. This can be done by analyzing the coefficients that are the ratios of intensity, i.e. such as pairs of diffraction lines I_{111}/I_{200}, I_{110}/I_{200}. The values of these coefficients for non-textured materials can be calculated from the data contained in international diffraction standards, i.e. ICDD cards [6]. Thus, when the ratio of intensities of two lines I_{h1k1l1}/I_{h2k2l2} for an experimental diffraction pattern is different from the same relation to the standard pattern, this means that the material is textured. The greater the difference between these values is, the stronger texture of the material is present at the material.

Another index is also applicable for assessing the texture degree. This is the ratio of the diffraction line intensity to the sum of the intensity of the i.e. first four lines obtained experimentally for the investigated sample to the same ratio for the standard diffraction pattern from the ICDD cards. For a non-textured material, this index equals one. The bigger the deviation of this index from value one is, the stronger is the material's texture.

When the analysis based upon the indexes discussed above shows that the material is textured, it is advisable to carry out a full texture analysis, however, this requires the use of complex measurement procedures.

According to the procedure described in the previous section, two texture indexes were calculated, namely:

- T_{hkl} – the ratio of the intensity of a particular diffraction line hkl next to the intensity of the strongest diffraction lines on the diffraction pattern
- M_{hkl} – the ratio of the experimental intensity of a diffraction line hkl next to the total intensity of the first four lines obtained for the samples to the same ratio for the standard pattern for non-textured material.

The results of these calculations are shown in Table 2 and compared with the values of texture indexes for material with random orientation (non-textured). On the basis of a comparison of texture indexes for the investigated samples with the indexes for a non-textured

TABLE 2
Texture indexes for two different areas of the samples from Inconel 718 in the initial state and after forging at 1100°C and 1000°C

Crystallographic plane			Random material		Initial material		After forging at 1100°C area1		After forging at 1000°C area1		After forging at 1100°C area 5		After forging at 1000°C area 5	
h	h	l	T_{hkl}	M_{hkl}	T_{hkl}	M_{hkl}	T_{hkl}	M_{hkl}	T_{hkl}	M_{hkl}	T_{hkl}	M_{hkl}	T_{hkl}	M_{hkl}
1	1	1	1.0	1.0	1.0	0.9	1.0	1.3	1.0	1.5	1.0	1.0	1.0	1.2
2	0	0	0.4	1.0	0.4	0.9	0.3	0.9	0.2	0.7	0.4	1.0	0.4	1.1
2	2	0	0.2	1.0	0.3	1.3	0.1	0.5	0.0	0.1	0.2	0.9	0.0	0.2
3	1	1	0.2	1.0	0.3	1.3	0.1	0.7	0.1	0.4	0.2	1.0	0.1	0.9
4	0	0	0.1	1.0	0.1	0.9	0.0	0.5	0.0	0.4	0.0	0.6	0.0	0.7

material, it is concluded that the material in the initial state either has a random orientation, or its texture is negligible. After forging, the texture degree increases. The highest texture degree is observed for the material after forging at 1000°C comparing to 1100°C especially at the area 5 (Table 2 – texture index M_{hkl}).

A lattice parameter was measured for the investigated material in the initial state and after forging. Lattice parameter can determined from the position of each single diffraction line separately. This method is not accurate and leads to results that slightly differ in value. The precise determination of the lattice parameter is based upon the procedure of Nelson-Riley (N-R) [11]. The precise calculation of the lattice parameter values is based on the linear interpolation of the lattice parameter values dependence on the Nelson-Riley function and on the equation describing this relationship. Upon this basis, the extrapolation and setting (of) the lattice parameter values is done for f(NR)=0, which corresponds to the diffraction angle of Θ =90° – Fig. 10. This way, the most precise lattice parameter is calculated with the accuracy of 0.001Å.

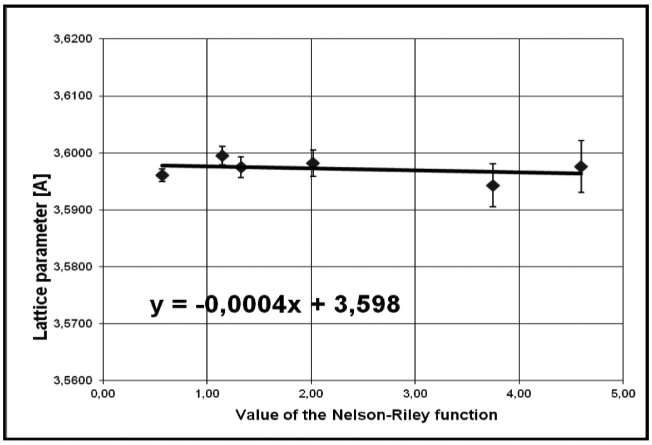

Fig. 10. Example of the N-R procedure – interpolation of the dependence of the lattice parameters on the value of the Nelson-Riley function and its extrapolation to the angle of Θ =90° corresponding to the value f (NR)=0

The value of the lattice parameters is important for the further study of the alloy, since it depends on the contents of elements in an alloy and can, therefore, be a measure of their concentration. The contents of these elements in the alloy affect the size of the lattice parameter and cause the shift of diffraction lines. In this way, it is possible to indirectly measure the contents of alloy elements dissolved in the solution. This is information about the form of the occurrence of alloy elements in the solution and it is also important for the evaluation of the material's properties. Using the described above Nelson-Riley procedure lattice parameters were calculated for all investigated materials – Tab. 3.

TABLE 3
Lattice parameters for material after thermo-mechanical treatment

Material	Lattice parameter [A]
Forging at 1100°-B1	**3,5987**
Forging at 1100°-B5	**3,5983**
Forging at 1000°-B1	**3,5980**
Forging at 1000°-B5	**3,5980**

The differences between lattice parameters are very small. A slightly higher values for material deformed at 1100°C indicate that the structure is more homogenous and also a little more alloying elements are dissolved at the γ (Ni-Fe) phase.

4. Conclusions

On the basis of the described investigation and study, the following conclusion can be presented:

1. After plastic deformation, slight changes in the phase composition of Inconel 718 alloy are observed. The dominant phase in the alloy is nickel by iron supersaturated.

2. Material forged at 1100°C is completely recrystallized by dynamic recrystallization, at 1000°C recrystallization occurs by creation of the new recrystallizated grains near or on the grain boundaries of existing grains.
3. After forging the texture is created at the deformed materials. Samples deformed at 1000°C have exhibited stronger texture comparing to these forged at 1100°C.
4. The strength of the texture is varied in samples cross-section, and the texture is stronger in the more deformed areas. Texture variation can have a significant impact on a material's property.

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REFERENCES

[1] A. Thomas, M. El-Wahabi, J.M. Cabrera, J.M. Prado, High temperature deformation of Inconel 718, Journal of Materials Processing Technology **177**, 469-472 (2006).
[2] H.Y. Zhang, S.H. Zhang, M. Cheng, Z.X. Li, Deformation characteristics of δ phase in the delta processed Inconel 718 alloy, Materials Characterization **63**, 49-53 (2010).
[3] H. Yuan, W.C. Lui, Effect of the δ phase on the hot deformation behavior of Inconel 718, Materials Science and Engineering A **408**, 281-289 (2005).
[4] B. Mikułowski, Stopy żaroodporne i żarowytrzymałe – nadstopy, Wydawnictwo AGH, Kraków (1997).
[5] P. Szablewski, M. Kawalec, Nierówności powierzchni obrobionej stopu Inconel 718 po procesie toczenia, Zeszyty naukowe Politechniki Poznańskiej (2005).
[6] H. Lu, X. Jia, K. Zhang, C. Tao, Fine-grained pretreatment process and superplasticity for Inconel 718 superalloy, Materials Science and Engineering A **326**, 382-385 (2002).
[7] M. Sundararaman, P. Mukhopadhyah, S. Banerjee, Precipitation of the δ-Ni_3Nb phase in two nickel base superalloy, Metallurgical Transactions A **19A**, 453-456 (1988).
[8] S.H. Fu, J.X. Dong, M.C. Zhang, X.S. Xie, Alloy design and development of Inconel 718 type Alloy, Materials Science and Engineering A **499**, 215-220 (2009).
[9] W.C. Lui, F.R. Xiao, M. Yao, Z.L. Chen, Z.Q. Jiang, S.G. Wang, The influence of cold rolling on the precipitation of delta phase in Inconel 718 alloy, Scripta Materialia **37**, 1, 53-57 (1997).
[10] A. Bunsch, M. Witkowska, J. Kowalska, Ocena własności strukturalnych materiałów polikrystalicznych metodami dyfrakcji rentgenowskiej XRD na przykładzie stopów Inconel 718, SIM 2011-s. 182.
[11] B.D. Cullity, S.R. Stock, Elements of X-Ray Diffraction, Prentice Hall, 2001.
[12] UNS N07718, W. Nr. 2.4668.

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