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On the accuracy of the X-ray diffraction quantitative phases analysis method in Inconel 718

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Inconel 718, a widely used Nb-modified nickel-base superalloy, consists of quite a few phases in the matrix such as δ, γ', γ'' and NbC, as well as small amounts of α-Cr, TiC, NbC, TiC, etc. Because of the determinant effects of the content of δ, γ' and γ'' phases in the microstructure on the mechanical properties of this alloy, many efforts have been focused in the aspect of quantitative phase analysis [1–4]. In our previous study [5], a simple X-ray diffraction quantitative phase analysis method for Inconel 718 has been proposed based on the relationship between the lattice constant of the γ phase and the content of precipitates, but the accuracy of this method has not been studied in detail. In this work, quantitative analysis was performed using X-ray diffraction method and selective electrolysis method, and the results were compared directly.

The composition of as-received Inconel 718 is shown in Table I.

Specimens under five different conditions were used. Condition A: solution at 970 °C for 1 h, a.c., then cold rolled to 40% reduction in thickness followed by a standard double aging (720 °C, 8 h + 620 °C, 8 h). Condition B: solution at 970 °C for 1 h, a.c., then cold rolled to 40% reduction in thickness, solution at 970 °C for 1 h, a.c., again cold rolled to 40% reduction followed by the double aging. Condition C: solution at 970 °C for 1 h, ac, cold rolled to 25% reduction in thickness, then solution at 970 °C for 1 h, a.c., again cold rolled to 25% reduction followed by the double aging. Condition D, E: sampled from different positions of a turbine disc after forging, then solution at 970 °C for 1 h followed by the double aging.

The samples for XRD quantitative analysis were mechanically polished followed with chemical etching. Quantitative analysis was performed in *D*/max-rB X-ray diffractometer with Cu K_α radiation. In detail see [5].

For the chemical quantitative analysis, extracting procedures of δ, γ' and γ'' phases from the matrix via selective electrolysis are as following. Extracting precipitates of γ', γ'', δ and NbC from the matrix via selective electrolysis under the conditions of (1.0% ammonium sulfate—2.0% tartaric acid—water) electrolyte, 0.025–0.03 A/cm^2 (current density) and below 5 °C. Then transferring the precipitates to a solution (5.0% vitriolic acid—5.0% tartaric acid), constant temperature bath 100 °C/1 h dissolving γ' and γ'' phases, while δ and NbC are retained. Similarly, under the conditions of (1.0% ammonium sulfate—3.6% zinc chloride—5.0% hydrochloric acid—methyl alcohol) electrolyte, 0.05–0.1 A/cm^2 and −5 °C, the NbC and δ-Ni_3Nb (indefinite quality) are extracted, then transferring precipitates into (1:1) vitriolic acid solution disaffiliating for 2.5–3 h, as a result the NbC remains. So far, the phases ($\gamma' + \gamma''$), δ and NbC are isolated, respectively [6].

TABLE I Chemical compositions of Inconel 718 (wt%)

C	Ni	Cr	Nb	Mo	Ti	Al	Mn	Si	P	S	Fe
0.04	52.52	18.34	5.10	3.07	1.00	0.50	0.02	0.11	0.004	0.002	Bal.

The isolated precipitates are identified using a Philips APD-10 X-ray diffractometer with Co radiation. Element contents of the extracted and isolated precipitates are measured via inductively coupled plasma-atomic absorption spectrometer (ICP-AAS).

By using anode selective electrolysis method, it is impossible to separate the γ' and γ''. But their content can be calculated by using the chemical compositions of ($\gamma' + \gamma''$) determined all together and those of γ' and γ'' determined individually by other methods. The latter ones can be found in [7], in which the chemical compositions of γ' and γ'' were determined individually by using an electron probe. The chemical compositions of ($\gamma' + \gamma''$) determined in our work and those of γ' and γ'' given in [7] are all listed in Table II. According to these data, the values of weight content of γ' and γ'' can be calculated, and the quantitative phase analysis results are listed in Table III.

From the X-ray diffraction pattern of Inconel 718, the presence of NbC, δ and γ phases can be clearly identified from the diffraction peaks. However, the (112) diffraction peak of γ'' phase is very close to the (111) diffraction peak of γ phase and the individual diffraction peaks of γ' phase cannot be observed because of its special crystal structure. In terms of the X-ray quantitative phase analysis method proposed previously [5], three equations were obtained by the comparison of integral areas of diffraction peaks. For the other two equations, one was conducted based on the relationship between the lattice constant of contents γ phase and the phases contents, and the other was proposed as $W_{\gamma''}/W_{\gamma'} = 4{:}1$. From the five equations the contents of different phases or their ratio can be calculated

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TABLE II Chemical compositions of ($\gamma' + \gamma''$) and those of γ' and γ'', at.%

Phase	Conditions	Nb	Al	Ti	Fe	Cr	Mo	Co	Ni	Remark
($\gamma' + \gamma''$)	A	14.03	6.23	6.42	2.99	3.62	0.66	0.82	69.23	Determined by
	B	13.52	6.28	6.13	2.85	3.71	0.64	0.56	70.32	chemical method
	C	9.35	6.17	6.38	2.40	4.71	0.74	0.03	70.23	
	D	11.96	3.99	5.98	1.79	3.27	0.72	0.04	71.77	
	E	11.76	4.2	6.3	1.64	3.95	0.67	0.04	71.43	
γ'		9.80	9.27	8.42	1.96	0.49	0.28		69.79	Average values
γ''		23.4	0.85	4.99	1.61	1.26	0.922		66.96	according to [7]

TABLE III Weight contents of different phases in Inconel 718, wt%

			Determined by XRD method		Errors of XRD results relative to chemical analysis, %	
Phase	Conditions	Determined by chemical method	According to $W_{\gamma''}/W_{\gamma'} =$ 4:1	According to $W_{\gamma''}/W_{\gamma'} =$ 3:1	According to $W_{\gamma''}/W_{\gamma'} =$ 4:1	According to $W_{\gamma''}/W_{\gamma'} =$ 3:1
($\gamma' + \gamma''$)	A	11.98	13.20		10.2	
	B	11.45	12.33		7.7	
	C	11.30	11.85		4.9	
	D	12.30	12.38		0.7	
	E	11.58	12.20		5.4	
γ'	A	3.09	2.64	3.30	−14.6	6.8
	B	2.88	2.47	3.08	−14.2	6.9
	C	2.95	2.37	2.96	−19.7	0.3
	D	3.09	2.48	3.10	−19.7	0.3
	E	2.95	2.44	3.05	−17.3	3.4
γ''	A	8.89	10.56	9.90	18.8	11.4
	B	8.57	9.86	9.25	15.1	7.9
	C	8.35	9.48	8.89	13.5	6.5
	D	9.21	9.90	9.28	7.5	0.8
	E	8.63	9.76	9.15	13.1	6.0
γ''/γ'	A	2.88				
	B	2.97				
	C	2.83				
	D	2.98				
	E	2.92				
δ	A	3.88	3.66		−5.7	
	B	5.35	5.75		7.5	
	C	5.43	5.91		8.8	
	D	3.22	3.40		5.6	
	E	3.40	3.52		3.5	
NbC	A	0.25	0.27		8.0	
	B	0.26	0.26		0	
	C	0.21	0.23		9.5	
	D	0.27	0.25		−7.4	
	E	0.26	0.26		0	
α-Cr	A	0.30				
	B	0.28				
	C	0.75				
	D					
	E					

directly, and the analysis results are also listed in Table III.

It can be seen that the quantitative results of ($\gamma' + \gamma''$), δ and NbC according to chemical analyses and those according to XRD analyses coincide with each other in most cases. But for the quantitative results of γ' and γ'' phases, large errors can be found for the XRD method relative to the chemical results. It has been widely accepted that the weight fraction of γ'' is four times bigger than the γ', but it is not the case in the present chemical analysis results($W_{\gamma''}/W_{\gamma'}$ is approximately 3). If we define the ratio of γ'' to γ' as 3 and use this relationship in the XRD analysis process, the quantitative results will be different (see Table III). According to the present relationship $W_{\gamma''}/W_{\gamma'} = 3$, the relative errors of the XRD quantitative results of γ' and γ'' phases are normally lower than 10%.

The chemical results indicate that the ratio of γ'' to γ' in Inconel 718 is approximately 3 for different treatment conditions. Introducing the relationship of $W_{\gamma''}/W_{\gamma'} = 3$ to the previously proposed XRD quantitative phase analysis method for Inconel 718, the results will be more accurate.

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