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Plasma assisted nitriding of Ni-based superalloys with various microstructures

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ABSTRACT

Ni-based superalloys are composed of a matrix with an austenitic face-centered cubic (FCC) crystal structure (γ phase) which is generally strengthened by the presence of a high fraction of ordered FCC precipitates γ' ($\text{Ni}_3(\text{Ti,Al})$ type) and/or γ'' (Ni_3Nb type). Plasma assisted nitriding of various Ni-based superalloys, either polycrystals (Haynes®230, Inconel®718, Udimet®720Li, N18) or single crystals (MC2 and MC-NG), presenting from 100 vol.% to 30 vol.% of γ phase, has been performed at moderate temperature (400 °C) to shed light on the specific responses of the different (δ , γ' , γ'') precipitates under nitriding. Characterizations by glow discharge optical emission spectroscopy and scanning electron microscopy have evidenced the formation of nitrided layers with thicknesses varying from ~1 μm to ~8 μm depending on alloys and treatment duration. In all alloys, X-ray diffraction on nitrided samples confirms that the γ phase is nitrided, as in austenitic stainless steels, to form the expanded austenitic FCC phase γ_N , a nitrogen insertion solid solution, with a mean concentration of ~25 at.% N. CrN nitrides were also identified. The incorporation of nitrogen within the precipitates appeared to depend on the alloy microstructure: for polycrystals, regardless of their proportion, the δ , γ' or γ'' precipitates seem nitrided in similar proportion than the matrix γ although the exact nature (nitrides or expanded δ , γ' , γ'' phases) of the obtained nitrided phases was not confirmed. On the contrary, in the single crystal MC2, the γ' precipitates accommodate a much lower nitrogen amount, in the range 3–5 at.%. Whatever the materials, the surface swelling induced by the nitrogen uptake is only relative to the total incorporated nitrogen quantity and is roughly independent on the nature of the nitrided phases.

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1. Introduction

Thanks to their good mechanical properties, nickel-based superalloys are widely used in pressurized water heat exchangers or in the hottest sections of aeroengines or industrial gas turbines, where they are subjected to high temperature and severe mechanical solicitations [1,2]. Superalloys typically have a matrix with an austenitic face-centered cubic (FCC) crystal structure called γ phase [3,4]. In addition to the hardening by substitution solid solution of the γ phase, some Ni-based superalloys gain their impressive mechanical properties from the presence of a high fraction of ordered $L1_2$ face-centered cubic γ' ($\text{Ni}_3(\text{Ti,Al})$ type) and/or tetragonal DO_{22} γ'' (Ni_3Nb type) precipitates [1–6]. However, increasing their resistance to very aggressive conditions (wear, fretting, oxidation, corrosion, higher temperatures and pressures, higher mechanical solicitations...) is still challenging in order to enhance the life time and to fulfill the expected future conditions of operations of the next generation gas turbines. As most of the limitations are coming from the surface behavior of the material, researches in surface treatments are worthwhile in conjunction with (bulk) material development.

Nitriding (gas nitriding, plasma nitriding, glow discharge, ion implantation ...) improves the mechanical, tribological or corrosion resistances of Ni-based superalloys. When performed at “high” temperature (>450 °C), the “internal nitriding” leads to more or less fine nitride precipitates responsible for the strengthening of the nitrided layer [7–17]. Chromium nitrides are mainly formed as Cr is usually the major nitride forming element in the studied alloys. However, when they are present in significant amounts, Ti, V or Nb can form nitrides too, which can provide a higher hardness than CrN [18,19]. The nitrided layer formed by moderate temperature (<450 °C) nitriding of Ni-based superalloys is usually composed of the expanded austenitic face-centered cubic phase, a nitrogen insertion solid solution called γ_N or S-phase, like in austenitic stainless steels (ASS) or CoCr alloys [20]. This nitrided phase usually shows higher hardness and gets improved tribological properties, without the loss of corrosion resistance, which can be interesting for applications keeping the superalloys at moderate temperature [1,13,15,18,21–27]. The expanded phase is metastable and decomposes into nitrides at high temperature [20]. The nitriding below 450 °C may however be an interesting way to introduce nitrogen in the materials, in order to obtain, after annealing process, specific structures and properties that cannot be achieved by direct high temperature nitriding.

Most of the previous studies are relative to solid solution strengthened Ni-based materials, i.e. without γ' or γ'' strengthening precipitates

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(like in, e.g., INCONEL® 600, 601, 690; NiCr, Nichrome...). Williamson et al. [23] and Eliassen et al. [26] studied the influence of several alloying elements in different Ni-based alloys. Elements in solid solution in the γ matrix like V, Nb, Cr, Ta, Al and Ti, as powerful nitride formers, were shown to promote the nitrogen uptake although, on the other hand, they destabilize the expanded phase towards the nitride formation. The significantly lower nitrogen uptake observed in the Nimonic 100 was ascribed to the amount of γ' precipitates [26]. By transmission electron microscopy, Xie et al. observed a complicated microstructure in Incoloy 908 implanted with nitrogen at 400 °C: in addition to some “degeneration” of the initial spherical γ' precipitates morphology (~30 nm in average diameter), dark spots are observed, likely relative to nitride precipitates or defect clusters [21]. In most of the previous studies where structural precipitates are present in the Ni-based superalloys, their influence and their specific behavior during the nitriding were not clarified and the superalloys were more or less considered as homogeneous materials [13,18,23]. In a recent paper [28], the γ' precipitates of the MC2 single crystal superalloy treated at 400 °C were shown to be almost non nitrided (<3 at.%), whereas the γ phase gave rise to an expanded γ_N phase, similar to the one obtained in stainless steel.

In this work, the nitriding at 400 °C of several Ni-based superalloys with various compositions and amounts of precipitates was performed; the study aims merely to determine whether the different precipitates (i.e. γ' , γ'' or δ) get nitrided as the γ phase does. The influence of these precipitates as well as of the composition of the different superalloys on the nitriding efficiency is beyond the scope of this work.

Polycrystals and single crystals were used and the γ proportion was varying from 100 vol.% to 30 vol.%. Thus six superalloys were nitrided at low temperature to determine the behavior of the different types of precipitates. In conjunction with usual chemical and microstructural characterizations, the surface swelling induced by the nitrogen uptake in the FCC matrix was used as an additional indicator of the nitrogen incorporation [29]. After a presentation of the main characteristics of the Ni-based superalloys in their as-received state (composition and volume fraction of the different phases), the modifications induced by the nitriding treatment are then given and discussed as a function of the γ' , γ'' or δ precipitate volume percentage.

2. Experimental details

2.1. Materials

The main characteristics of the different alloys are summarized in Tables 1, 2 and 3. Haynes® 230 (Ha230), Inconel® 718 (IN718), Udimet® 720Li (U720) and N18 are polycrystals while MC2 and MC-NG are single crystals. Ha230, IN718, and U720 are cast and wrought alloys while N18 is a powder metallurgy one. It should be noted from Table 2 that direct aged IN718 samples have been used in the present study (i.e. without any solution heat treatment). In the different polycrystalline superalloys, the γ phase grains are of few tens or hundreds of μm size. Table 3 shows the typical volume proportion and composition of each phase gathered from literature. Considering the applied thermal treatments (Table 2), these data can be considered roughly valid for the studied samples as only the main differences between γ

Table 1
Compositions of the different alloys in at.% (main elements only).

Material	Ni	Cr	Fe	Mo	W	Co	Mn	Al	Ti	Nb	Ta	Re	Ru
Ha230	Bal.	26.5	3.4	1.3	4.8	5.3 ^a	0.6	0.7	0.1				
IN718	Bal.	20.5	23.3	1.7		1.0	0.4	0.4	0.8	2.9			
U720	Bal.	17.3		1.8	0.5	14.1		5.2	5.9				
N18	Bal.	12.3	0.1	3.8		14.8		9.1	5.1				
MC2	Bal.	9.3		1.3	2.6	5.1		11.2	1.9		2.0		
MC-NG	Bal.	4.7		0.6	1.7			13.6	0.6		1.7	1.3	2.4

^a Maximum.

Table 2
Heat treatments and average grain sizes of the alloys (AQ = air quench).

Material	Solution heat treatment	Aging heat treatments	Average size of the γ matrix grains (μm)
Ha230	5 min at 1232 °C/AQ	/	50
IN718	/	8 h at 720 °C, cooling at 50 °C/h 8 h at 620 °C, AQ 24 h at 700 °C, AQ + 16 h at 815 °C, AQ	7–15 20–700
U720	4 h at 1155 °C, AQ	24 h at 700 °C, AQ + 4 h at 800 °C, AQ	15
N18	4 h at 1165 °C, AQ	6 h at 1080 °C, AQ + 20 h at 870 °C, AQ	Single crystal
MC2	3 h at 1300 °C, AQ	4 h at 1100 °C, AQ + 16 h at 870 °C, AQ	Single crystal
MC-NG	3 h at 1310 °C, heating at 3 °C/h, 3 h at 1340 °C, AQ		

phases and (γ' , γ'' or δ) phases will be discussed in this article. More precise distinction between the various precipitates in the different materials would have required additional detailed analysis.

Ha230 can be here considered as a “reference” material because it consists of 100 vol.% of γ phase, without any precipitate. The (M_6C ($\text{Ni}_3\text{W}_3\text{C}$) and M_{23}C_6 ($\text{Cr}_{21}(\text{W},\text{Mo})_2\text{C}_6$) carbides in Ha230 will not be considered due to their low proportion [30]. In the other materials, the volume fraction of the γ phase then decreases down to 30 vol.% with few intermediary points at 75, 60 and 45 vol.%. Most of the chromium in the different alloys is found in the γ phase, with a concentration higher than 22 at.%. The γ phase in IN718 contains a significant amount of Fe whereas the γ phase in U720 and N18 is rich in Co. MC-NG is significantly different with only 11 at.% of Cr in the γ phase thanks to the high content in Re and Ru.

U720 and N18 alloys contain a multimodal γ' distribution with primary, secondary and tertiary precipitates (γ'_{I} , γ'_{II} , γ'_{III} respectively) of various sizes [31,32]. In addition to a small amount of γ' , IN718 contains γ'' and δ (the thermodynamically stable Ni_3Nb phase, with orthorhombic structure) precipitates and few primary (Nb,Ti)C and secondary (M_{23}C_6) carbides [33]. Single crystalline MC2 and MC-NG alloys have only one type of precipitates (γ'_{I}) whose size is close to 0.35–0.5 μm [34,35]. The Cr, Fe and Co concentrations in these γ' , γ'' and δ precipitates do not exceed 5 at.% whereas they contain almost all the Al, Ti and Nb components. It should be noticed that the elemental composition of the γ' precipitates depends on the investigated materials (see references in Table 3). In the polycrystals the Al and Ti contents of the γ' precipitates are in the range 8–13 at.% and 9–14 at.%, respectively. For the MC2 and MC-NG single crystals, the γ' precipitates have a higher Al content (15–17 at.%) and lower Ti or Ta content (<3 at.%). This means that they are closer to the “perfect” intermetallic Ni_3Al compound.

Table 3
Typical volume fraction (in vol.%) and typical chemical compositions (in at.%) of the different phases in the different alloys as compiled from the literature [50–54].

Alloys	Phase	vol. %	Ni	Cr	Fe	Co	Al + Ti + Ta + Nb	Other elements
Ha230	γ	100	Bal.	26.5	3.4	5.3 ^a	0.8	6.7
IN718	γ	75	Bal.	22.0	23.8		3.2	2.9
	γ'	4	Bal.	0.5	2.1		27.6	0.5
	γ''	21	Bal.	0.8	0.9		30.4	1
	δ		Bal.	3.4	5.3		24.2	2.2
U720	γ	55–60	Bal.	34.6		17.6		3.5
	γ'	40–45	Bal.	2.8		5.0	23.9	2.7
N18	γ	40–45	Bal.	25.7	0.2	25.3	2	8.4
	γ'	55–60	Bal.	1.6		6.6	21.5	2.2
MC2	γ	30	Bal.	25.9		9.1	2.6	6.9
	γ'	70	Bal.	1.8		3.5	17.9	3.7
MC-NG	γ	30	Bal.	11.3			3	12.8
	γ'	70	Bal.	2.0			17.8	4.5

^a Maximum.

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