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Development of a TiC_p Reinforced Ni-Based Superalloy MMC, with High Creep Resistance and Reduced Weight

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Abstract. Ni-based superalloys, in both single and polycrystalline varieties, are extensively used in high pressure turbine blades. But contrary to single crystal variants, the polycrystalline forms present easier manufacturing and offer higher potential for improvement in metal matrix composites (MMCs). To benefit from this opportunity, an Inconel X-750 superalloy reinforced with TiC particles is proposed, having a polycrystalline microstructure and the possibility for weight reduction in turbine elements application. The metallic powder with an addition of 15 vol.% of 3.7 μm_d TiC particles was prepared through low energy mixing, uniaxial pressing and sintering, followed by a triple heat treatment. The microstructure was analyzed with SEM and XRD techniques. Compressive creep tests were performed at 800 °C with 200 MPa, on both original and reinforced alloys. The study shows how the inclusion of a highly compatible particle reinforcement does not only improves the creep resistance, but also reduces the material weight, thus having potential to promote further reduction in the creep rate on turbine blades submitted to centripetal forces.

Introduction

Metal matrix composites (MMCs) are known for combining some of the best properties of two materials classes; metals and ceramics. Particle reinforced MMCs, specifically, have been studied for their excellent isotropic creep resistance, making these materials suitable for multiaxial loaded applications at high temperatures. MMCs frequently also present lower density, creating opportunities for weight reduction in components used in transportation, energy and industrial infrastructure, thus enhancing the energy efficiency in these applications. Among them, aircraft engines and industrial turbines are envisaged as potential targets for the use of MMCs in blades and discs [1,2].

Extensive work has been done with lightweight MMCs, notably with the use of aluminum matrices. Such materials may present yield strength (YS) up to 500 MPa, and Young's Modulus up to 100 GPa, when adding 20 vol.% of Al_2O_{3p} as reinforcement [2]. However, to obtain even better mechanical properties, simply increasing the volume of particles is not without consequences. After a certain limit, not only the ductility decreases sharply – resulting in elongation values below 1% – but often also the creep resistance worsens [3].

To surpass this limitation, a material with superior resistance is required for the matrix, as well as a highly compatible and stiff reinforcement. This paper presents a concise overview on particle reinforced MMCs, in the context of creep resistance, and proposes the development of a new Ni-based Inconel X-750 superalloy reinforced with titanium carbide particles (TiC_p), comparing it to the non-reinforced alloy.

Matrix and reinforcement selection

Ni-based superalloys are vastly used in turbine components, for their high creep and corrosion resistance. While the polycrystalline form is common in aircraft engines in general, having equiaxial or columnar grains, the single crystal variant is preferred for high-pressure blades in modern aircrafts equipped with large turbofan engines [4]. But despite presenting the highest creep resistance among all Ni-based superalloy forms, they are also high-priced and offer limited room for improvements, thus creating opportunity for the further development of polycrystalline forms with the addition of stiff ceramic particles.

Though the reinforcement by particles does not benefit remarkably from the load bearing capability, when compared to fiber-reinforced MMCs, it is advantageous for multiaxially loaded systems and provides easier manufacturing processes. The reinforcement materials are chosen with respect to their interaction with the matrix, including wetting capability, formation of interfacial products, density, thermal expansion coefficient and Young's modulus, among others [2].

The particle inherent Young's modulus is intended to be significantly higher than the matrix one, since the reinforcing phase is expected to bear the maximum possible load without cracking. Damaged particles not only are less effective to carry load, but depending on their shape they can also produce voids, being highly detrimental to the MMC Young's modulus and creep life [5]. It is also known that larger particles are the first to suffer damage during creep [6–8].

A small difference in the thermal expansion coefficient between matrix and reinforcement is also usually beneficial, by inducing tensile and compressive stresses in the system during temperature changes. They can raise the dislocation density in the particles vicinity, acting as an additional strengthening mechanism. The effect is stronger when smaller particles are used as reinforcement [9,10].

Among potential candidates to reinforce nickel-based superalloys, titanium carbide (TiC) combines a relative low density (4.93 g/cm³), high melting point (3338 K) and increased Young's modulus (350 GPa at 973 K) [11]. TiC also possess good binding with pure Nickel, presenting a wetting angle of 30° (at 1723 K under vacuum) [12], a value that can be significantly improved by alloying [2]. Furthermore, inherent (Nb,Ti)C particles are formed in several aged Ni-based superalloys, such as the Inconel X-750. Experiments with TiC_p added to pure Ni and to other Inconel alloys did not show significant dissolution of the reinforcement phase, or the formation of intermetallic phases at the particle/matrix interface [13,14].

While TiC_p can be considered generally stable in Ni-based superalloys, it is also known that a reaction with Nb might occur at elevated temperatures. During cooling from 1423 K, a MC-type precipitate – rich in Nb and Ti – is the first to be formed in a non-reinforced Inconel X-750. It is also speculated that the NbC carbides possess higher stability in Ni-based superalloys, when compared to TiC [15]. While preparing by arc-melting a mixture of pure Nb powder, TiC powder and sponge Ti, Wei *et al.* (2009) observed the formation of (Nb,Ti)C and Nb_2C carbides [16]. Similarly, when depositing pure Nb powder on a cast iron substrate by laser melting, da Costa *et al.* (2004) observed the formation of Nb_2C. The precipitate remained stable even after long isothermal treatment at 1023 K [17].

Volume, shape and size of reinforcing particles

For most structural applications, it is common to limit the volume of reinforcement to less than 30% in the MMC design. These materials retain some ductility and manufacturing capacities of the original alloy, having particles predominantly surrounded by the metallic matrix. But while in general the addition of a higher vol.% of reinforcement brings important benefits, increasing the stress exponent and reducing the minimum creep rate [18–20], it is also found that after reaching an upper limit of vol.%$_p$ – in the range of 20 to 40 vol.% – the creep life is severely shortened [21,22]. This is attributed to the formation of a particle network, leading to a situation where the reinforcement phase is not completely surrounded by the matrix anymore, but instead forming large clusters. During creep, the plastic deformation in the clusters zone promotes stress concentration, setting off the early formation of cracks [3].

The reinforcing particles can be nearly spherical, or possess any other geometrical form, provided that the aspect ratio between their axes length is kept close to 1. It is shown [5], theoretical and experimentally, that discontinuous reinforcements with an aspect ratio further from 1, such as whiskers and platelets, yield a stronger contribution to raise the dislocation density during deformation, when compared to equiaxed particles. However, if sharp edges are present, they may contribute to the premature formation of cracks, diminishing the MMC life under creep. Furthermore, cracks can also originate within the reinforcement itself, through preexisting inner defects, or from particles that suffered damage during the plastic deformation. If the damage results in acute shaped fragments, these will also act as stress concentrators, contributing to lower the creep resistance [23].

It is shown that increasing the particle size often leads to a lower general strengthening effect. Moreover, the life under creep may also significantly diminish when increasing the particle size, with early failure caused by a stronger tendency to cavitation [5,24]. Oppositely, decreasing the particle size below a certain value – in the range of tens of nanometers – leads to the loss of its load-bearing capability. For particles below this size the reinforcement main strengthening mechanism shifts to the impediment of moving dislocations, and the material is more frequently labeled as a dispersion reinforced metal, rather than a MMC [1,25].

Materials and methods

Powders of Inconel X-750 (composition given in Table *1*) with granulometry d_{80} = 22 µm, and 99 wt.% pure TiC, with d_{80} = 3.7 µm, were mixed through a low energy process. The combined powder was uniaxially hot pressed and sintered under vacuum at 1523 K and 35 MPa for 1 h, in the form of a squared plate. A triple heating treatment was applied, comprised of solutioning (4 h at 1423 K), stabilization (24 h at 1115 K) and precipitation (20 h at 977 K), always with the use of air cooling between steps. Cylindrical specimens of 5 x 7.5 mm (d x h) were machined out of the plate and submitted to compressive creep tests at 1073 K (800 °C), with a stress of 200 MPa.

The same heat treatment, machining and compressive tests were applied to a commercially available Inconel X-750, provided as extruded bars. Microstructural analysis was conducted with light and scanning electron microscopy (SEM), energy dispersive spectrometry (EDS) and X-ray diffraction (XRD).

Table 1 - Chemical composition (wt.%) of Inconel X-750 powder.

Element	Cr	Fe	Ti	Nb	Al	Si	Mn	Co	C	Cu	S	N
Wt. %	15.3	6.5	2.71	1.1	0.7	0.2	0.1	0.03	0.02	0.01	0.003	bal.

Results

Light microscopy results of the non-reinforced alloy revealed substantial scattering in grain size, with a mean diameter of 107 ± 67 µm (Figure *1*a). Grain boundaries exhibited lenticular-shaped precipitates, measuring < 1 µm, while bigger, differently shaped precipitates measuring 1-10 µm were found dispersed on both grain boundaries and grain interiors. The TiC_p-reinforced alloy presented less grain size scattering, with a mean diameter of 9 ± 4 µm (Figure *1*b). MMC grain boundaries displayed TiC particles in both isolated and clustered forms.

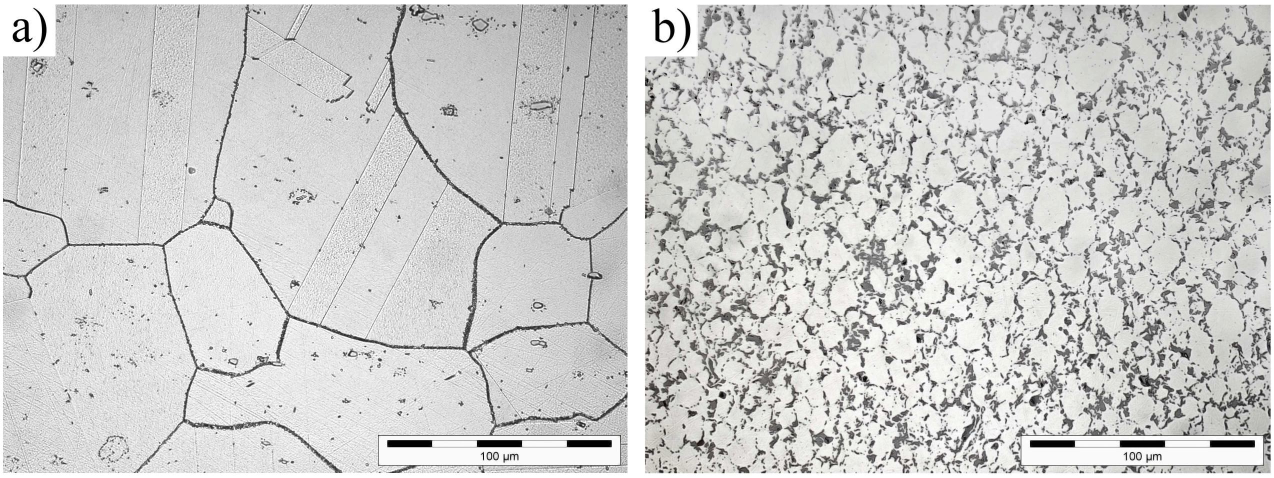

Figure 1 - Light microscopy of Inconel X-750 after heat treatment, in the conditions: a) non-reinforced; b) reinforced with TiC particles. Etching: H_2MoO_4 + HCl. Magnification: 500x.

Both original and TiC_p reinforced alloys showed the presence of (Nb,Ti)C in SEM images (Figure *2*) and punctual EDS analysis, as inherent MC precipitates and added reinforcing particles, respectively. Additionally, chromium-rich $M_{23}C_6$ precipitates were also identified at grain boundaries of both materials. Gamma prime (γ') precipitates were not seemingly affected by the TiC added particles in the MMC samples, maintaining similar morphology and size.

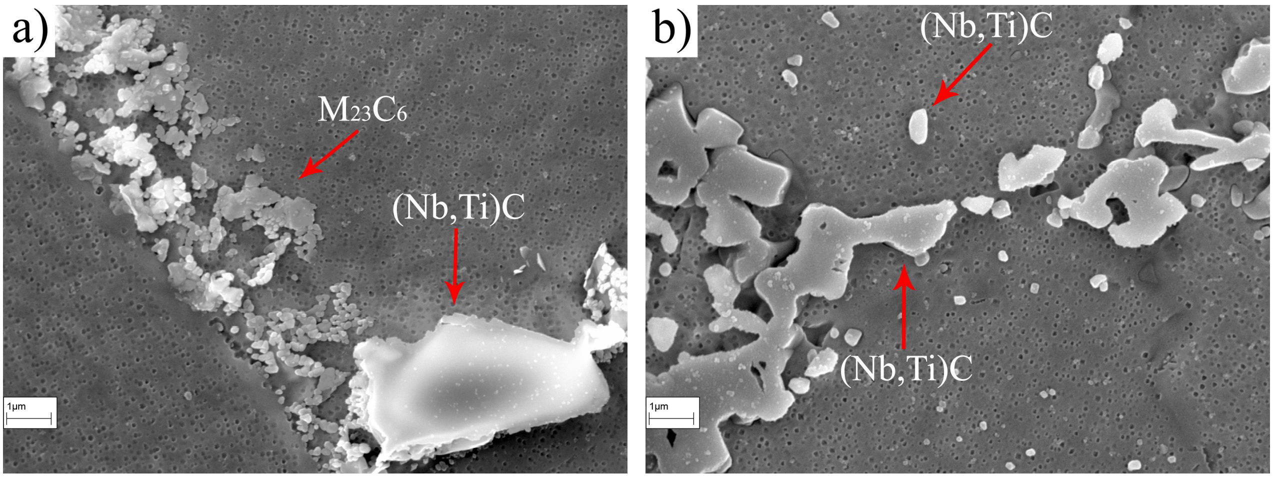

Figure 2 - Scanning electron microscopy of Inconel X-750 after heat treatment, in the conditions: a) non-reinforced; b) reinforced with TiC particles. Etching: H_2MoO_4 + HCl. Magnification: 20.000x.

Element mapping by EDS revealed in the MMC that all the reinforcing TiC particles were enriched by Nb (Figure *3*), notably in their peripherical areas. These particles were devoid of Ni, Fe and Cr, but in a few cases presented a higher concentration of Al. The distribution of γ' precipitates in the matrix, however, remained unaffected also in the vicinity of these areas.

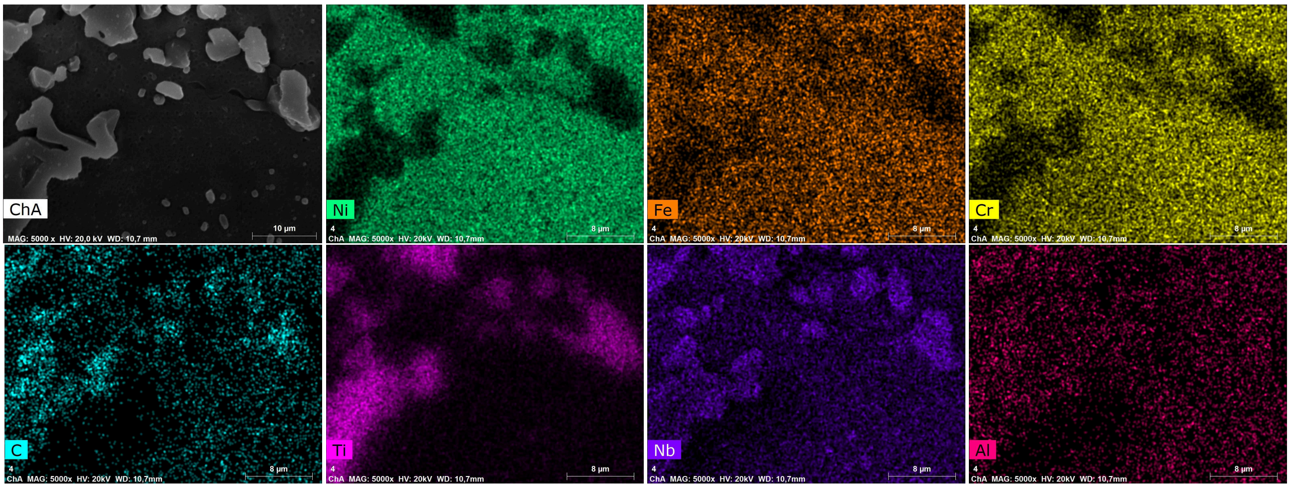

Figure 3 - EDS elemental mapping showing the TiC particles added to the alloy, with the reinforcing phase enriched with Nb after the sintering process.

All the expected phases in the Inconel X-750 after the triple heat treatment were identified with XRD on both original (Figure *4*a) and TiC_p-reinforced (Figure *4*b) alloys, namely γ (Ni), γ' (as Ni_3Al), $M_{23}C_6$ (as $Cr_{23}C_6$) and MC (as NbC). The original material presented additionally $AlNbNi_2$, while in the MMC were also identified $Al_3Ti_{0.75}Fe_{0.25}$ and the added TiC phase.

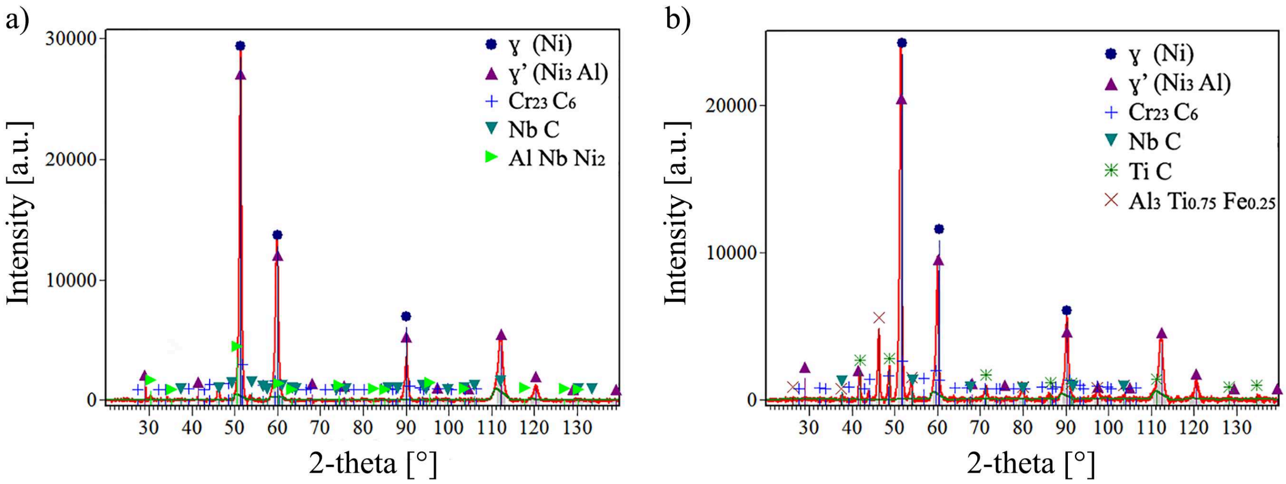

Figure 4 - XRD diffractograms of both original (a) and TiC_p-reinforced Inconel X-750 (b) alloys.

Compressive creep tests were terminated after the tertiary stage was reached, at 70 and 150 h for original and reinforced alloys, respectively, representing an increment of 114% in steady-state creep for the composite. The creep rate, measured at the secondary stage, was 1.35×10^{-8} s^{-1} for the Inconel X-750, and 4.58×10^{-8} s^{-1} for the MMC (Figure 5). To achieve 1% of strain – a common creep limit desired in turbine blades [26] – 15,5 h and 47,3 h were required for the original and reinforced alloys, respectively, resulting in an improvement of 205% for the MMC.

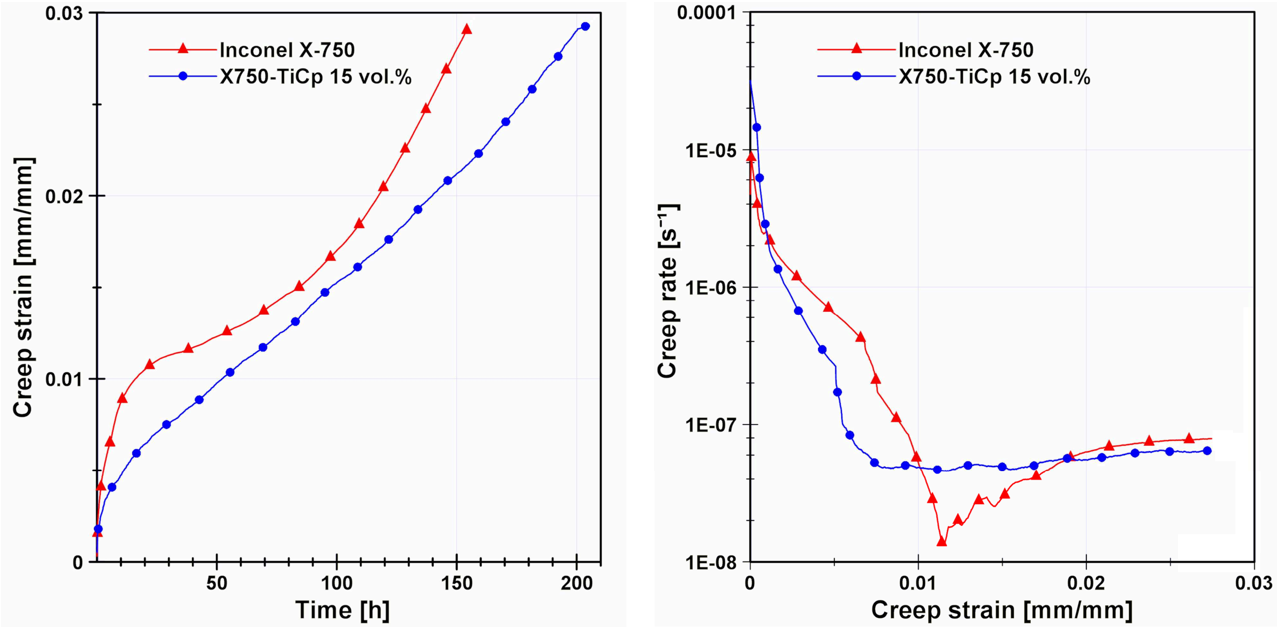

Figure 5 - Compressive creep curves for both original and reinforced alloy, in tests conducted at 1073 K (800 °C) with 200 MPa.

Discussion

When compared to the original alloy, the reinforced material presented a much finer grain structure, due to the boundaries nucleation along the fine TiC particles around the matrix powder. This arrangement led to the undesired formation of clusters, but also reduced the concentration of chromium carbides in grain boundaries, once these were further dispersed in the finer microstructure. Furthermore, the reinforcing TiC particles were enriched by Nb presented as solute in the matrix, thus approaching the composition of the (Nb,Ti)C precipitates inherent in the Inconel X-750 and providing evidence of the affinity between reinforcement and matrix.

The initial compressive creep test results showed improvement of the MMC over the original alloy. But concerning the testing type, it is necessary to take into account possible differences when considering future applications. Contrary to tensile creep tests, that produce voids during the secondary creep stage, compressive creep tests do not lead to active damage (open microcracks) in isotropic metals [27]. However, a good agreement was found by Zhu *et al.* (1998) between tensile and compressive results of a Ti/TiCp 15 vol.% MMC, obtaining similar strain rates in both tests at 873 K (600 °C) under 100 to 400 MPa [28]. Nevertheless, it has to be proofed first that tensile and compressive creep test results correspond for the proposed MMCs.

Despite both materials presenting creep rates in the same order of magnitude, with marginally slower rates for the original alloy, it is noteworthy that the non-reinforced material also required considerable less time to achieve 1% of strain and to enter the tertiary stage of creep, when compared to the composite. The stability of the secondary creep stage is essential to preserve the material function, avoiding critical dimension changes in turbine elements, for instance. Furthermore, the MMC exceeded 2% strain at the test conditions before entering the tertiary creep stage, exhibiting significant ductility at 1073 K (800 °C) for the envisaged applications.

Regarding the proposed composite use in turbine blades, it is known that many MMCs present a threshold stress, leading to higher than expected stress exponent (n) values [29,30]. The following equation is commonly used to analyze the strain rate in the secondary creep stage, where A and K are constants, σ is the applied stress, σ_{th} is the threshold stress, T is the temperature in Kelvin, Q' is the activation energy and n' is the stress exponent:

$$\dot{\varepsilon} = A(\sigma - \sigma_{th})^{n'} exp\left(-\frac{Q'}{KT}\right) \quad (1)$$

When combining the higher creep resistance with a density reduction in the MMC, and comparing it to the non-reinforced alloy, it is possible to obtain an exponential gain in the creep rate reduction on turbine blades submitted to centripetal forces. Considering the equation (*1*), once the weight reduction is proportional to the stress reduction on a cross section of the blade, the higher stress exponents present on MMCs have the potential to further affect the creep rates in several orders of magnitude.

Conclusions

- A cost-effective Inconel X-750/TiC_p 15 vol.% MMC, with improved creep resistance – when compared to the non-reinforced alloy – was proposed for use in aircraft engines and industrial turbines.
- The MMC microstructure exhibited evidence of good affinity between the added TiC particles and matrix, with the formation of NbC in the reinforcing phase, notably over the particles surface.
- Initial compressive creep results showed significant improvements on the MMC creep resistance over the original material at 873 K (600 °C), with longer times to reach 1% of strain (205% gain) and tertiary creep stage (114% gain). The higher stability in the secondary creep stage is an important aspect for turbine elements, which rely upon strict dimensional control to adequately perform under high stresses and temperatures.
- When combined, the MMC higher intrinsic creep resistance and material lower density show the potential to reduce creep rates by several orders of magnitude, as a result of lower centripetal forces acting on a turbine blade section.

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