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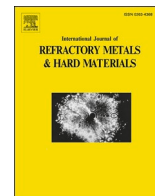
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Powder HIP of pure Nb and C-103 alloy: The influence of powder characteristics on mechanical properties

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ABSTRACT

In this work, pure Nb processed by Powder Metallurgy Hot Isostatic Pressing (PM HIP) was investigated as a potential alternative to C-103 Nb-based alloy for the manufacture of near-net-shape thruster combustion chambers. Three Nb powders with varied particle size range (fine, mid-range and coarse) and C-103 powder were investigated to understand the differences in particle size distribution (PSD), morphology and oxygen (O) content present in the alloy chemistry. The as-HIPed microstructures of pure Nb and C-103 are characterised by a near to fully dense microstructure and the absence of PPBs. Microstructural analyses performed on pure Nb highlighted the influence of particle size on the average as-HIPed grain size. Additionally, it was observed that O content plays a crucial role in the microhardness of pure Nb. Tensile tests performed on Nb mid-range and on Nb mid-range sieved showed that a simple sieve operation was effective in increasing the strength of the material while maintaining good levels of elongation. Alternatively, it was also demonstrated that pure Nb powder heat treatment can be regarded as an effective way to increase the O levels in pure Nb powders as witnessed by the high hardness levels. On the other hand, as-HIPed C-103 powder showed superior tensile properties if compared to the minimum specifications for wrought C-103. Finally, to summarise, the work performed on pure Nb, a simple structure-property relation model was developed to predict the YS of pure Nb based on the O levels and grain size of the as-HIPed microstructure.

1. Introduction

Nb alloys are widely used in the aeronautic and space sectors due to their ability to retain good mechanical properties at considerably high temperatures [1–5]. Over the years, different types of Nb alloys have been developed including solid solution strengthened Nb alloys (Cb-132, F-48, SCb-291 and SCb-298) and dispersion strengthened Nb alloys (Cb-65, D-12, Nb–1Zr and C-103). Nb alloys are mostly characterised by the presence of Hf, W, Mo, Ta, Zr and Ti to improve the strength and high-temperature oxidation resistance capabilities [5,6]. The main strengthening contribution in Nb alloys comes from the substitutional solid solutions of Mo, W, Ta and V [6,7]. Precipitation hardening is also an important strengthening mechanism in Nb alloys, made possible by the

addition of interstitials, such as C, N and O, with the presence of reactive elements such as Zr, Ti or Hf.

Among the different Nb alloys, the most commonly used Nb alloy for thruster's combustion chambers is C-103 (Nb-10Hf-1Ti) [7]. As stated above, the role of Hf, Ti and Zr in C-103 is to react with the interstitials, especially O and C to generate stable oxides and carbides responsible for the increase in high-temperature strength ($>0.5T_m$) and improvement of high-temperature oxidation resistance [3,5,6]. However, the use of C-103 alloy has some limitations related to cost, procureability, welding and coating. In fact, the presence of 10% Hf makes C-103 difficult to weld due to the formation of brittle intermetallics, and it is responsible as well for the generation of cracks and coating spalling after repeated thermal cycling. Additionally the use of conventionally manufacturing

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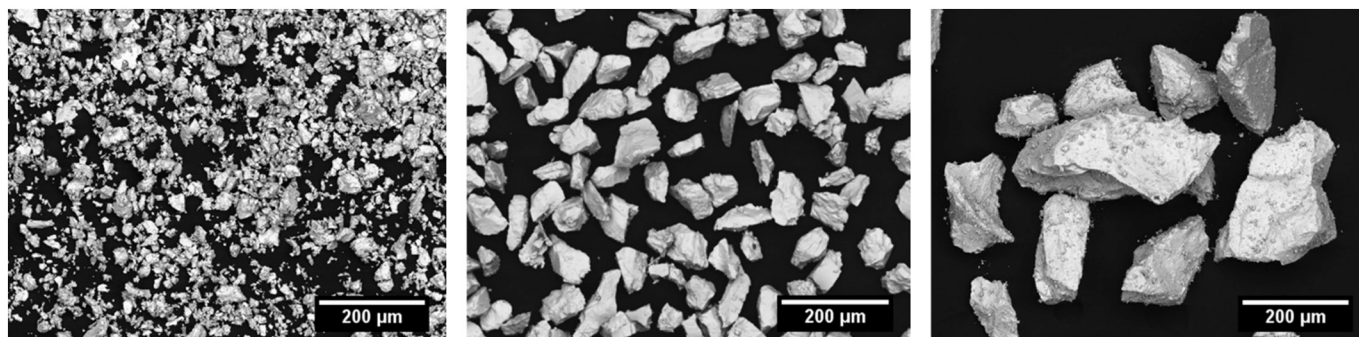


Fig. 1. SEM backscattered micrographs of Nb fine, mid-range and coarse powders.

routes such as casting and forging lead to a considerable material waste resulting in a highly inefficient and costly process. The use of near net-shape hot isostatic pressing (NNS HIP) for the manufacture of thruster combustion chambers can mitigate the raw material cost and the lead times associated with machining, by drastically reducing the buy-to-fly ratio, and consequently the amount of machining required. However, the presence of elements with high affinity to O such as Hf, Zr and Ti can represent a challenge. In fact, O and C present during atomisation process is responsible for the formation of oxides and carbides at the prior particle boundaries (PPBs) in the as-HIPed microstructure, especially in highly alloyed materials with the presence of reactive elements such as Hf, Ti, Zr, Cr and Al [8,9]. This will drastically reduce the mechanical properties of the material, especially in terms of ductility and fracture toughness [10].

On the other hand, pure Nb, due to the absence of strong oxide formers and as a consequence of high O solid solubility limit is perfectly suitable for PM HIP with no formation of PPBs. In fact, if during the HIP process O levels are below their solid solubility in Nb, the formation of discrete oxides at PPBs during the HIP process will be prevented. Additionally, the use of reactive elements such as Hf, Ti and Zr in Nb alloys will reduce the oxygen solubility limits, consequently reducing the strengthening effect coming from the solid solution of oxygen in the Nb matrix [7]. Additionally, the use of pure Nb would guarantee a better metallurgical compatibility with the high-temperature oxidation-resistant coating with no formation of brittle intermetallics due to the absence of Hf. Therefore, the possibility of using the PM HIP technique for pure Nb to improve its mechanical properties was investigated in the current study. The influence of oxygen on the mechanical properties of pure Nb has been widely reported in literature [11–13]. In particular, Sankar et al. reported a considerable increase in strength for pure Nb by increasing the O levels from 50 to 800 ppm [11]. Cordero et al. have established the Hall–Petch parameters for pure Nb, finding that the O contribution to the solid solution strength for pure Nb is 0.19 MPa/ppm [12]. The conventional manufacturing routes, such as casting and

forging, do not provide the required flexibility in terms of process parameters and feedstock material to control the oxygen levels. PM HIP allows to easily manipulate the characteristics of the powders i.e. chemistry and particle size distribution, and consequently tailoring the microstructure and mechanical properties of the material. Furthermore, the commonly used manufacturing process for Nb powders is based on the Hydride–Dehydride (HDH) process, which is capable of producing powders with good control of the contamination levels [14].

2. Experimental

In this study, the HIP response of pure Nb powders with three different particle size ranges and a C-103 powder was investigated. The pure Nb powders were categorised as fine (<44 µm), coarse (<500 µm) and mid-range (15–100 µm) whereas C-103 (10–45 µm) powder was manufactured by argon gas atomisation (AGA) process.

A detailed powder characterisation was performed on the four powders including powder morphology, particle size distribution (PSD) and the determination of oxygen content present in the alloy chemistry. Powder morphology was assessed using a Hitachi TM3000 Scanning Electron Microscope (SEM). PSDs for the four powders were determined by laser diffraction method using a Malvern Panalytical Mastersizer3000 system. The chemical analyses of the four powders were performed by AMG Analytical Services, Sheffield. The Inductively Coupled Plasma Optical Emission Spectroscopy (ICP OES) technique was used to determine the major alloying elements in C-103 i.e. Nb, Hf, Ti, Zr, Ta and W. Whereas C was determined by LECO thermal infrared using combustion in oxygen; and O, N and H gases were determined by LECO inert gas fusion.

After characterisation, Nb and C-103 powders were encapsulated in low carbon steel (ISO 3574 Grade CR4) cylindrical canisters (Ø50mm x 90 mm) with 2 mm thickness. The filling procedure was performed in a glovebox to avoid any interaction with the external environment. After filling, the canisters were de-gassed for 48 h to a pressure in the order of

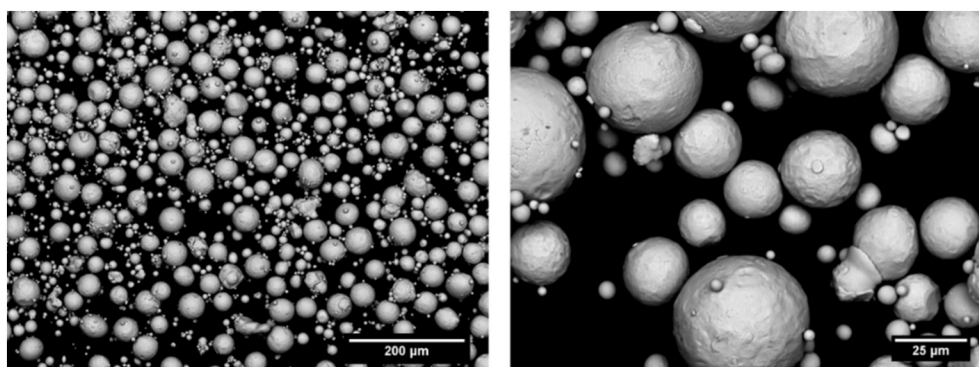


Fig. 2. SEM backscattered micrograph of C-103 powder.

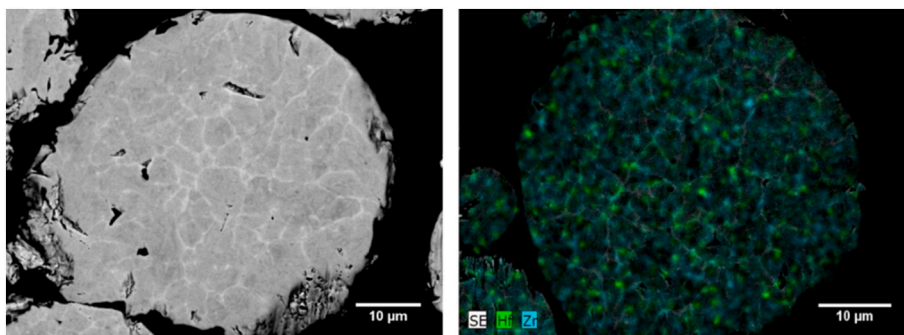


Fig. 3. SEM backscattered micrographs of C-103 powder cross section (left), C-103 powder cross-section EDS (right).

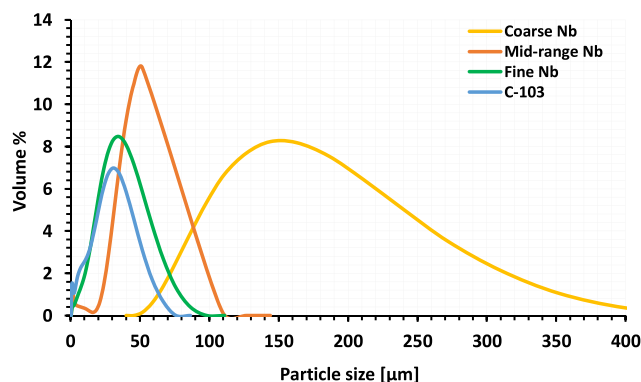


Fig. 4. Particle size distribution of Nb and C-103 powders.

10^{-5} mbar and hot sealed.

All the powder filled canisters were HIPed using EPSI HIP system installed at University of Birmingham. After HIP, the microstructural analysis samples were extracted, mounted in conductive Bakelite and polished using standard metallographic techniques. The samples' microstructure was then analysed using an optical microscope and SEM. Electron backscattered diffraction (EBSD) was performed on the three Nb samples using a Philips XL-30 SEM and the post-processing was performed using ATEX software [15]. To assess the oxygen pickup in pure Nb samples, inert gas fusion using a LECO system was performed on the as-HIPed Nb samples. The microhardness tests were performed on the HIPed powders according to ASTM E384–17, and by making 10 indents per sample, using a semi-automatic Buehler microhardness tester with a load of 300gf. The room temperature tensile tests were conducted on as-HIPed Nb powders according to ASTM E8/E8M–16a.

3. Results and discussion

3.1. Powder characterisation

The morphology of the three Nb powders is shown in Fig. 1. There is a considerable difference in the powder size and consequently in PSDs. Fine Nb powder is characterised by small particles surrounded by very fine ones, adversely affecting the flowability. Furthermore, the different powders all show a rock shape, which is typical of powders obtained by the HDH process. C-103 powder has a nearly spherical shape with the presence of some satellites and some irregular particles caused by the collision of molten metal with an already formed particle (Fig. 2). The C-103 powder cross section in Fig. 3 reveals the presence of Hf and Zr micro-segregation in the inter-cellular regions.

The PSDs in Fig. 4 show a considerable difference between fine and coarse powders, while less difference in particle size is observed between fine Nb and C-103 powders. Coarse powders show a bi-modal

Table 1
PSDs of Nb and C-103 powders.

Powder	D10 [µm]	D50 [µm]	D90 [µm]
Nb fine	7	29	57
Nb mid-range	3	50	82
Nb coarse	101	203	2050
C-103	2	20	46

Table 2
Oxygen content on Nb powders.

Powder type	Oxygen content [ppm]
Nb fine	1849
Nb mid-range	302
Nb coarse	1172

distribution with a D_{50} of 203 µm, higher if compared to fine and mid-range powders. Mid-range powders have a D_{50} of 50 µm, higher if compared to the D_{50} of 29 µm for fine powders (Table 1). The difference observed in powder characteristics will give a different HIP response in terms of macro-scale effects, such as shrinkage, which is dependent on the packing density. Most significantly the microstructure, such as grain size and consequently the mechanical properties of the material, will be highly affected by the powder characteristics.

As mentioned above, the O content is an important factor influencing the mechanical properties of pure Nb and C-103 powders. It determines an increase in strength for pure Nb, and adversely influences the mechanical properties of C-103 by increasing the oxide precipitation at PPBs. The oxygen content of the three Nb powders is reported in Table 2, while the chemical analysis performed on C-103 powders is shown in Table 3. The oxygen measurements highlight a considerable difference among the four powders. It is worth noting that the O levels are not directly linked with the particle size. Fine powders show very high oxygen concentration (1849 ppm); similar values are observed for coarse powders (1172 ppm); while mid-range Nb and C-103 powders show a much lower amount of oxygen, (302 ppm and 371 ppm, respectively). Nb powders have higher O levels if compared to commercial Grade 2 (250 ppm) [16], on the other hand, C-103 powder has lower levels of Zr if compared to the standards, while the levels of oxygen are higher than the requirements [17]. Higher O levels, compared with the levels indicated in the standards, will contribute to a drastic increase in the solid solution strengthening of the material, with the possibility of retaining good levels of ductility as highlighted in the work by Yang et al. [18].

The role of Zr in Nb alloys is to improve the creep resistance and high-temperature oxidation properties of the material by reducing the oxygen solid solubility in the alloy and thus promoting the formation of oxides [7]. However, the absence of Zr in the alloys does not compromise the room temperature mechanical properties for the alloy and can be beneficial for limiting the presence of PPBs due to the high affinity of

Table 3
Chemical analysis of C-103 powder (wt%).

Powder	Nb	Hf	Ti	Zr	Ta	W	C	O	N	H
Required [17]	87.3	9–11	0.7–1.3	0.7	0.5	0.5	<150 (ppm)	<225 (ppm)	<100 (ppm)	<15 (ppm)
C-103	89.08	9.35	1.07	<0.05	0.30	0.19	38 (ppm)	371 (ppm)	24 (ppm)	7 (ppm)

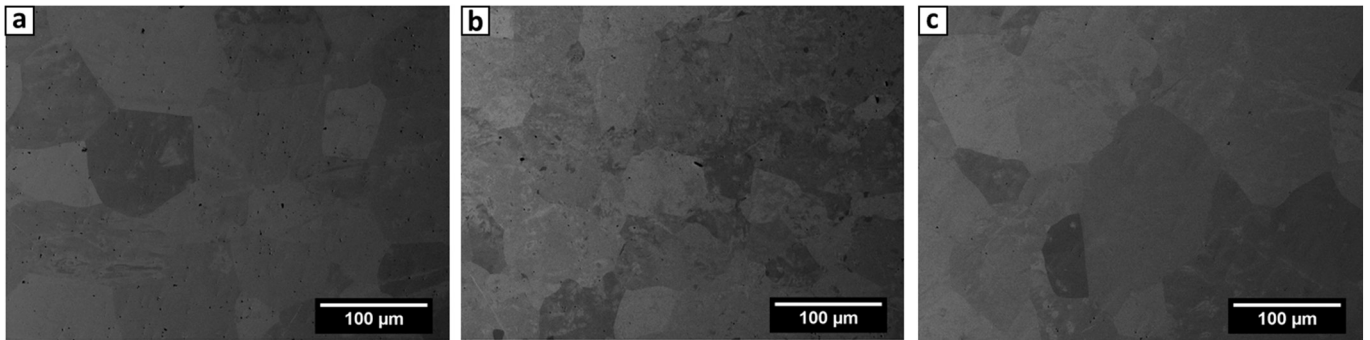


Fig. 5. Backscattered SEM micrographs of as-HIPed microstructure of (a) Nb fine, (b) Nb mid-range and (c) Nb coarse.

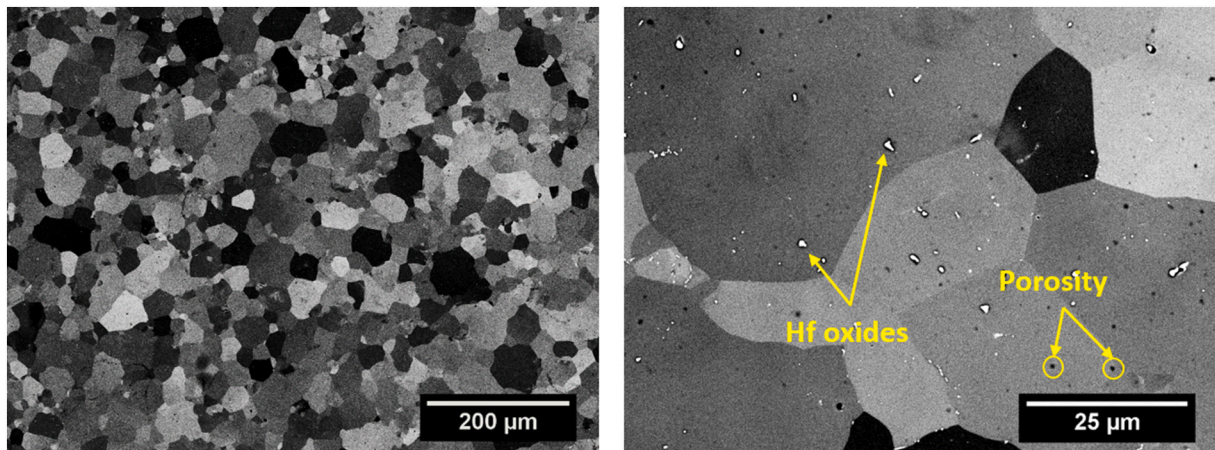


Fig. 6. Backscattered SEM micrographs of as-HIPed C103.

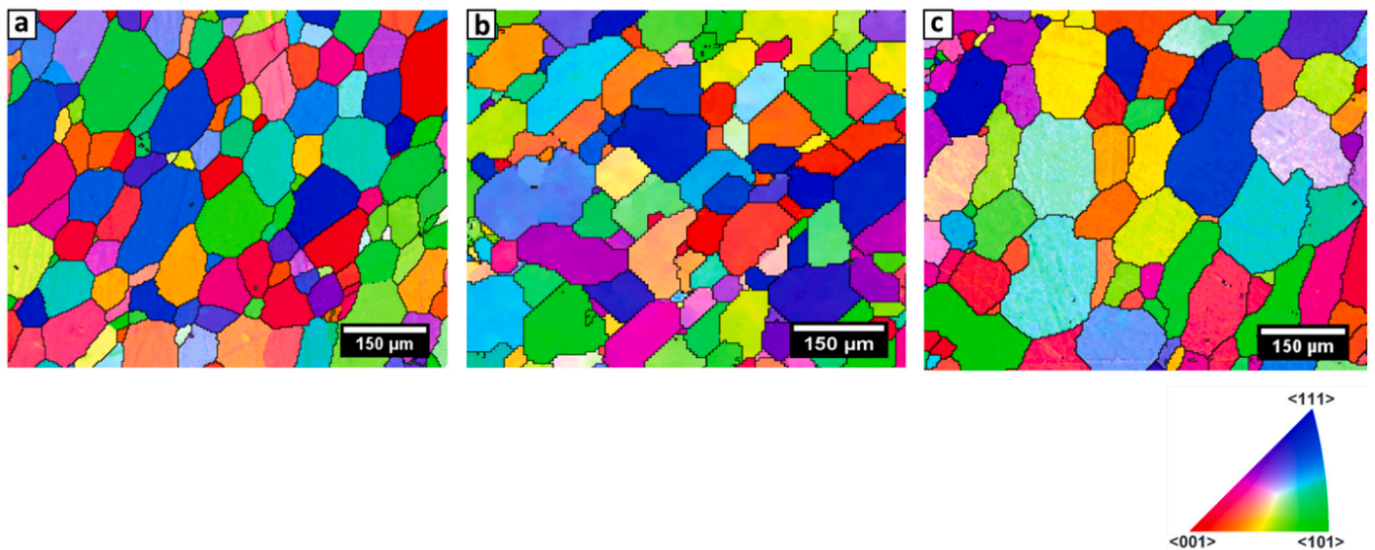


Fig. 7. EBSD of (a) Nb fine, (b) Nb mid-range and (c) Nb coarse.

Table 4
Oxygen measurements of as-HIPed Nb.

As-HIPed powder type	Oxygen content [ppm]	Oxygen pickup [ppm]
Nb fine	2361	512
Nb mid-range	390	88
Nb coarse	1438	266

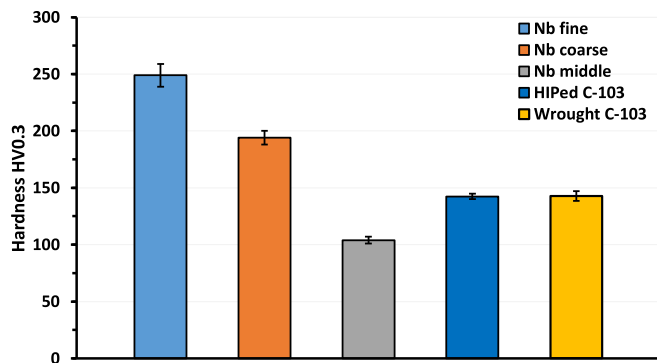


Fig. 8. Microhardness values of as HIPed Nb vs wrought C-103.

Zr to O.

3.2. Microstructure analysis

Backscattered SEM micrographs of the as-HIPed Nb samples are shown in Fig. 5. All three microstructures present equiaxed grains, typical for HIPed materials. Even in this case, some differences can be observed. As-HIPed fine Nb powders show a finer grain structure if compared to mid-range and coarse microstructures. Another important result to capture is that all three microstructures are near-fully dense. The microstructure of as-HIPed C-103 is shown in Fig. 6. The HIPed material presents some limited porosity and is characterised by the presence of some precipitates. The precipitates are rich in Hf and can be identified as Hf (O, C) [19]. The use of higher HIP temperatures would reduce the porosity in the as-HIPed C-103 samples. However, higher HIP temperatures can lead to eutectic reactions between the Fe present in the canister and the Hf contained in C-103 alloy, which happens between 1300 and 1400 °C. Additionally, higher HIP temperatures would promote excessive grain and precipitate coarsening in the as-HIPed microstructure.

EBSD analyses were performed on the Nb as-HIPed microstructures. Fig. 7 shows a randomly oriented structure with equiaxed grain size for the three HIPed Nb samples. The EBSD maps clearly show the presence

of a fully recrystallised microstructure free from PPBs for the three Nb HIPed powders. The absence of PPBs can be related to the high solid solubility of oxygen in Nb at the HIP temperature [7]. Another important outcome from the EBSD analysis is the differences in grain size among the three microstructures. Nb fine shows an average grain size of 44 μm; Nb mid-range shows an average grain size of 53 μm; while in Nb coarse the calculated average grain size was 67 μm, which is almost twice the size of Nb fine. The difference in grain size among the three powders is a function of the powder particle size (Fig. 4). In fact, Nb fine showed the smaller grain size, while Nb coarse is characterised by the biggest grain size. This is an important outcome because it demonstrates the possibility to tailor the as-HIPed mechanical properties of pure Nb by using different powder particle sizes. Furthermore, especially for pure metals, the large difference in grain sizes revealed by the EBSD maps can be directly linked to the mechanical properties of the material.

Oxygen measurements were also performed on the as-HIPed samples to outline the differences in O content, and ascertain the O pickup during the HIP procedure. Nb fine showed the highest level of O pickup, this can be attributed to the higher levels of surface to volume ratio enhancing the reactivity of the powder. Lower O pickup can be observed in coarse powders, while minimal O pickup is observed in mid-range powders (Table 4).

3.3. Mechanical properties

The four as-HIPed samples were tested using microhardness to have a first understanding of the mechanical properties of the material. The results in Fig. 8 show a large difference in hardness between the three Nb powders and surprisingly high levels of hardness if compared to HIPed C-103. Fine Nb powders show the highest hardness of 250 HV, while Nb mid-range shows the lowest hardness, just above 100 HV. These results highlight a direct correlation between the O-levels and the hardness of the material. The results also outline that the grain boundary strengthening is not the main strengthening mechanism if compared to the solid solution one. In fact, the HIPed Nb coarse powder has a considerably higher hardness than Nb mid-range, despite the bigger grain size. C-103 has a hardness of 142 HV much lower if compared to Nb fine and Nb coarse HIPed powders using the same HIP conditions. This suggests that by increasing the O-levels in pure Nb, it is possible to obtain higher room temperature strength if compared to as-HIPed C-103.

The first reason behind this behaviour is found in the preferential precipitation of oxide and carbides in HIPed C-103 at PPBs. Furthermore, the formation of Hf (O, C) will reduce the matrix solid solution strengthening by concentrating part of the Hf at PPBs. The other reason is found in the O-levels: C-103 has much lower O-levels if compared to Nb fine and Nb mid-range, the presence of lower O-levels in C-103 will reduce the oxygen solid solution strengthening, if compared to Nb fine

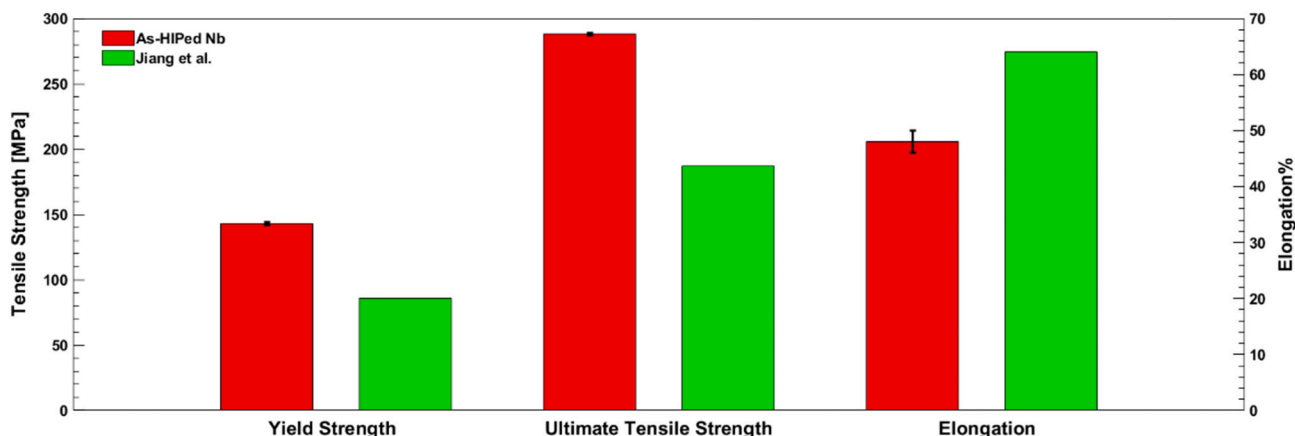


Fig. 9. Tensile properties of as-HIPed Nb mid-range [20].

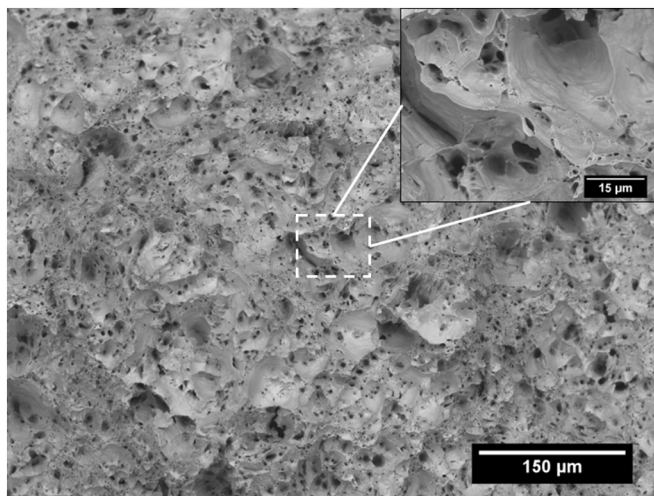


Fig. 10. Fractured tensile sample Nb mid-range.

and Nb coarse as-HIPed materials. However, in contrast to Nb, it is not advisable to increase the O-levels in C-103 powders due to the limited oxygen solid solubility in the alloy. Lastly, Fig. 8 shows that the hardness of wrought and annealed C-103 (courtesy of Nammo) is similar to the HIPed C-103, with the hardness level below fine and coarse HIPed Nb.

More detailed analyses are required to have a better understanding of the mechanical properties of the as-HIPed pure Nb and their comparison with HIPed C-103. To this end, mid-range Nb powders were used for tensile testing of Nb, for two main reasons. Firstly, because the wide range distribution could potentially give more flexibility in terms of an alteration of PSD and consequently powder characteristics, which is one of the objectives of this study. The second reason is linked to Nb mid-range lower oxygen level, which could give more information on the influence of oxygen variation on the mechanical properties of the as-HIPed Nb. The results for the room temperature tensile test on mid-range powders can be seen in Fig. 9. The tensile properties show a yield strength of 143 MPa and a UTS of 288 MPa, much higher if compared to the strength values reported in the literature [20]. As expected, the elongation was lower, but the value of 48% still represents an excellent result for the proposed application.

The higher strength reported for mid-range Nb powders is a combination of oxygen levels and fine grain size obtained using PM HIP. Some fractographic analyses were performed on the fractured tensile samples. The SEM micrographs clearly show a ductile fracture, where the main fracture mode is represented by micro void coalescence. It is important to note on the fractured surfaces that no presence of oxides acting as nucleation sites for fracture can be observed (Fig. 10).

As-HIPed Nb showed superior tensile properties if compared to wrought Nb, however, the tensile strength obtained is still below the required range for the application, and more importantly, still not comparable to Nb alloys commonly used in the space sector [7]. Thanks to the previous investigation performed on different powders, it is possible to understand the influence of powders' characteristics on the mechanical properties. Thus, it was decided to perform sieving on the mid-range powders to increase the surface to volume ratio of the powder to increase the oxygen concentration in the powder. Additionally, sieving will target to obtain a finer as-HIPed microstructure by reducing the average powder particle size, further contributing to an increase in strength. Sieving was performed using a 63 μm sieve in a protected atmosphere. Nb mid-range powder was selected for sieving operations because of its wider PSD (Fig. 4), allowing to further reduce the particle size by sieving operations. Additionally, both Nb fine and Nb coarse experienced high O-levels which would result in a drastic reduction in ductility.

After sieving, the oxygen content of the powder was checked to

Table 5

Oxygen levels of Nb mid-range powder before and after sieving.

Powder type	Oxygen content [ppm]
Nb mid-range	302
Nb mid-range sieved (63 μm)	756

understand if there is a relevant increase in the O-levels. Table 5 summarises the O-levels before and after sieving. It shows that just by powder sieving, the O-level increased from 302 ppm to 756 ppm. Once the increase in oxygen was confirmed, the sieved powders were HIPed using the same HIP parameters of the mid-range powders.

Fig. 11 highlights the microstructure of the sieved Nb powder, as expected the microstructure is characterised by the presence of a near-fully dense and equiaxed microstructure with an average grain size of 45 μm, smaller if compared to the as-HIPed Nb mid-range. Thus, even in this case, reducing the particle size resulted in a reduction of the average grain size in the as-HIPed microstructure. The combination of finer microstructure with a higher O-levels can result in a consistent increase in strength for as-HIPed sieved Nb powder if compared to as-HIPed Nb mid-range.

To check for a possible increase in strength, the sieved powders were then tested using microhardness. As expected, the microhardness results show an increase from 104 HV for mid-range HIPed sample to 135 HV for the sieved one. Tensile tests on the sieved mid-range powders have been performed to understand the response in terms of tensile strength and elongation for the material. The results presented in Fig. 12 show a noticeable increase in both YS (from 143 MPa to 200 MPa) and UTS (from 270 MPa to 350 MPa) while maintaining good levels of elongation, far above Nb alloys used for the same application [7].

The fractographic micrographs of Fig. 13 show a ductile fracture with the presence of dimples, similarly to the observations for HIPed Nb mid-range (Fig. 10). The room temperature tensile strength values are now much closer to the required values, further sieving can be performed to increase the strength to the required levels.

Room temperature tensile tests were also performed on as-HIPed C-103 to understand and compare the tensile properties with pure Nb, and with the specifications for the wrought alloy. C-103 tensile properties are highlighted in Fig. 14. The as-HIPed alloy shows excellent properties if compared to the minimum specification for wrought alloy both in terms of strengths and ductility. Furthermore, despite the possible formation of Hf-rich PPBs, C-103 shows superior room temperature properties if compared to the sieved mid-range Nb. Additionally, if compared to the work of Philips et al., as-HIPed C-103 is characterised by slightly higher levels of YS and UTS, accompanied by a lower ductility. This can be associated with the higher O-content in C-103 powder promoting precipitation of Hf-rich PPBs which impair the ductility [21].

The fractographical analysis presented in Fig. 15 shows a ductile fracture mode with the presence of Hf-rich oxides on the fracture surface, confirming the influence of PPBs on the tensile behaviour of HIPed C-103.

3.4. Influence of oxygen on mechanical properties

It was demonstrated that the O-content plays a crucial role in the mechanical properties of as-HIPed pure Nb. Harris et al. demonstrated the correlation between O-levels and hardness [22]. Their proposed equation is presented below:

$$HV = 69.5 + 881 O + 296 N + 271 C \quad (1)$$

where O, N and C are expressed in wt%. It is possible to see from Eq. 1 that the O-level has a big impact on the hardness, much higher if compared to N and C. The equation was plotted against the available data on HIPed Nb of the current study (Fig. 16). The values obtained in this study are in strong agreement with the empirical model, especially

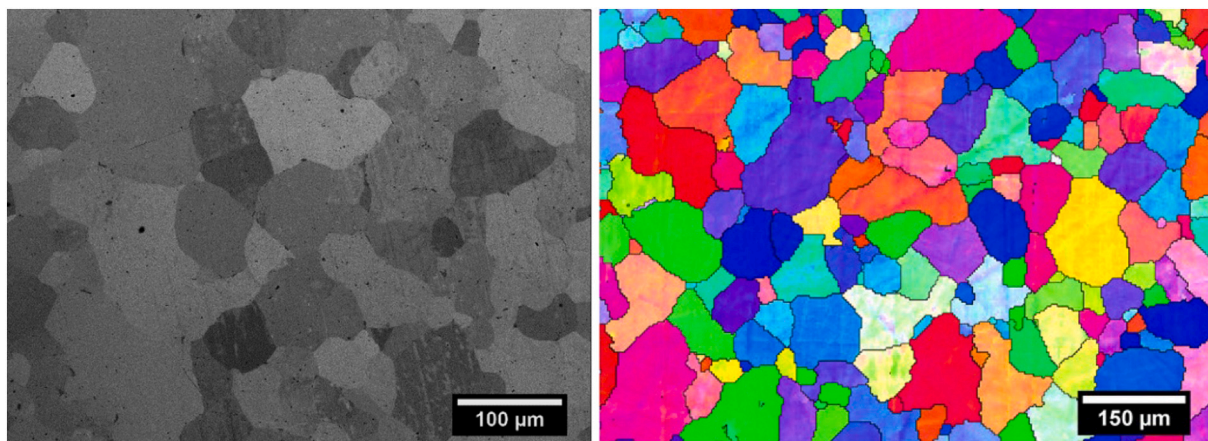


Fig. 11. SEM backscattered micrographs of as-HIPed sieved Nb powder (left), EBSD map of as-HIPed sieved Nb powder (right).

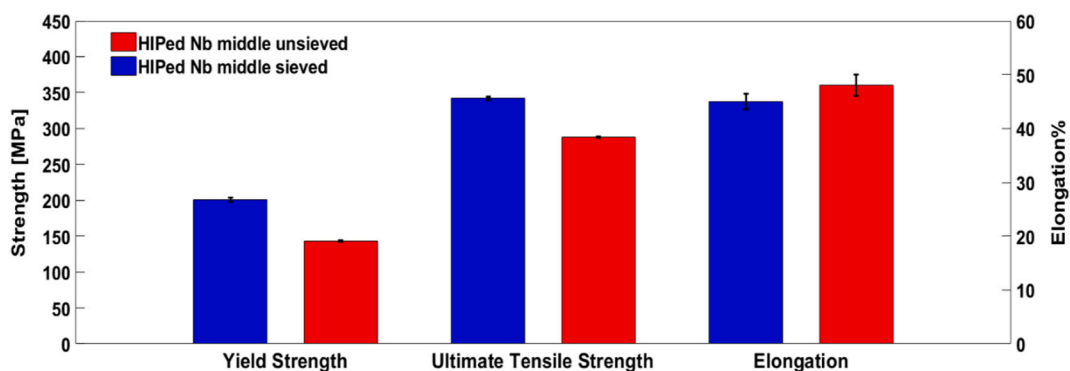


Fig. 12. Mechanical properties of as-HIPed Nb. Comparison between mid-range and sieved powders.

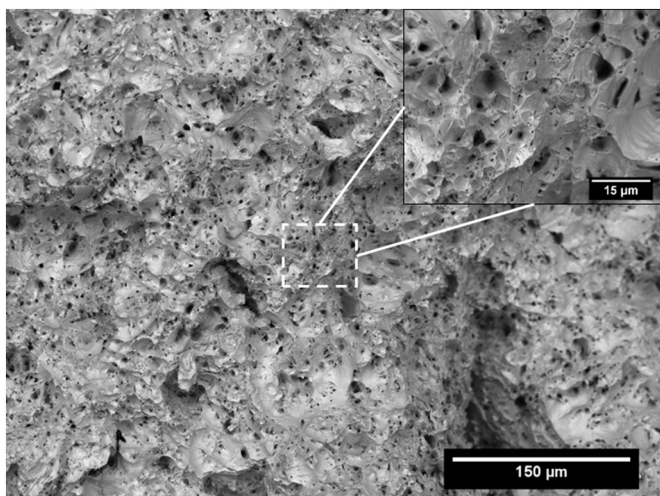


Fig. 13. Fractured tensile samples of as-HIPed sieved mid-range Nb.

for lower O- levels. The equation also indicates that to achieve the same hardness of C-103, an O-level of around 900 ppm is required for pure Nb.

In order to understand the combined effect of the O-levels and grain size on the strength, and calculate the O-level needed for achieving the required strength, a simple model was presented. This was based on the structure-property relation for the material, and on the coefficients available in the literature [12,23]. The general equation for structure-property relation is presented below (Eq.2) [24].

$$\sigma_{YS} = \sigma_0 + \sigma_{SS} + k_y d^{\frac{1}{2}} + \alpha G b \sqrt{\rho} \quad (2)$$

where: σ_{YS} = yield strength of the material. σ_0 = inherent lattice strength. k_y = strengthening coefficient. d = grain size. α = material parameter. b = Burgers vector. ρ = dislocation density.

The following assumptions were made on as-HIPed Nb:

- No presence of any precipitate. This assumption is true for pure metals, such as Nb. The presence of oxides was neglected because the O-levels are below the solid solution levels at HIP temperature [7,25].
- No dislocation strengthening. The microstructure during HIP evolves via recrystallisation, thus after HIP it can be assumed that the microstructure is free from dislocations.

The values for inherent lattice strength of Nb, k_y , n and σ_{SS} were obtained from [12].

The values for YS were calculated for both Nb mid-range and sieved Nb. The results presented in.

Table 6 shows a good agreement with the tested samples, with an error of just 1 MPa for mid-range Nb and an error of 25 MPa for sieved Nb.

Based on the above results, which give a good approximation of the YS for Nb powders, it was possible to calculate the O-level required to achieve the desired YS of 260 MPa. In this case, the O-level required to achieve that YS is 1000 ppm, assuming a grain size similar to as-HIPed Nb sieved.

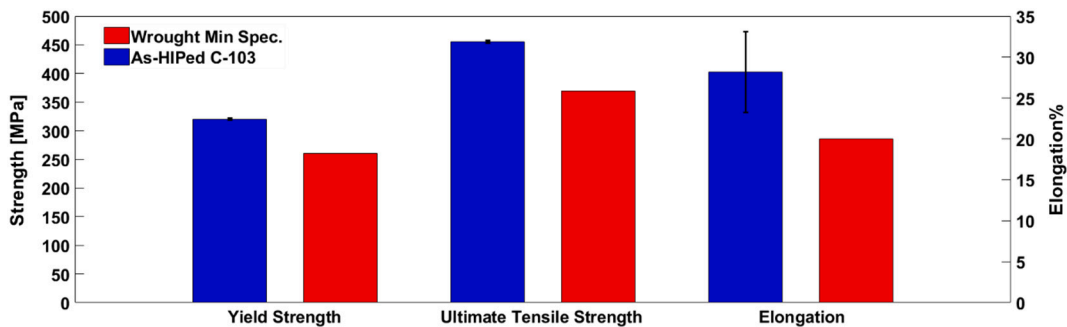


Fig. 14. Room temperature tensile properties of as HIPed C-103 vs ASTM minimum specifications for wrought C-103 [17].

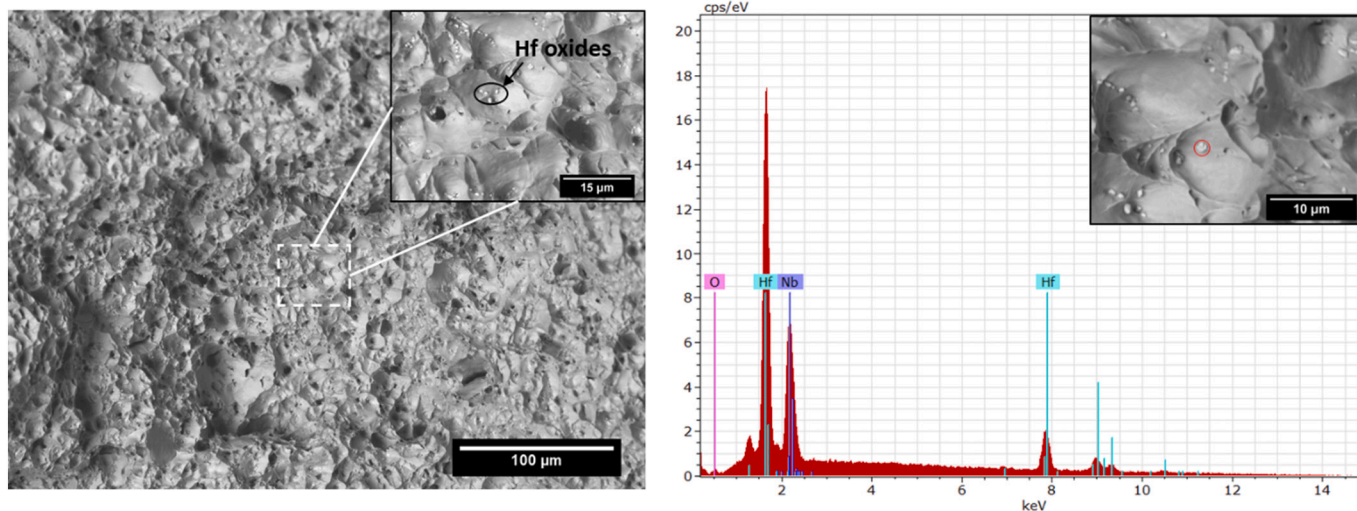


Fig. 15. Fractured tensile samples of as-HIPed C-103 (left), EDX spectrum of white particle on the fracture surface (right).

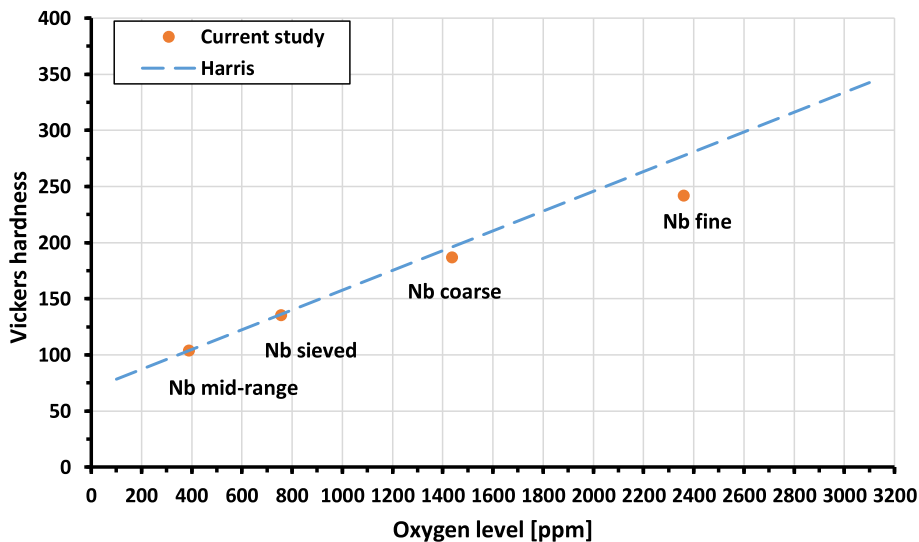


Fig. 16. Influence of oxygen on the hardness of pure Nb. Experimental values obtained in the current study vs Harris proposed Eq. [22].

Table 6
Experimental YS vs calculated YS for mid-range and sieved HIPed samples.

	Experimental YS	YS model prediction
Nb mid-range	143 MPa	142 MPa
Nb sieved	201 MPa	226 MPa

3.5. Powder heat treatment

The abovementioned sieving strategy was capable of increasing the O-level and, consequently, the strength of the material. However, the oxygen strengthening contribution of the powder is not enough to match the required strength. The possible solution proposed was to perform

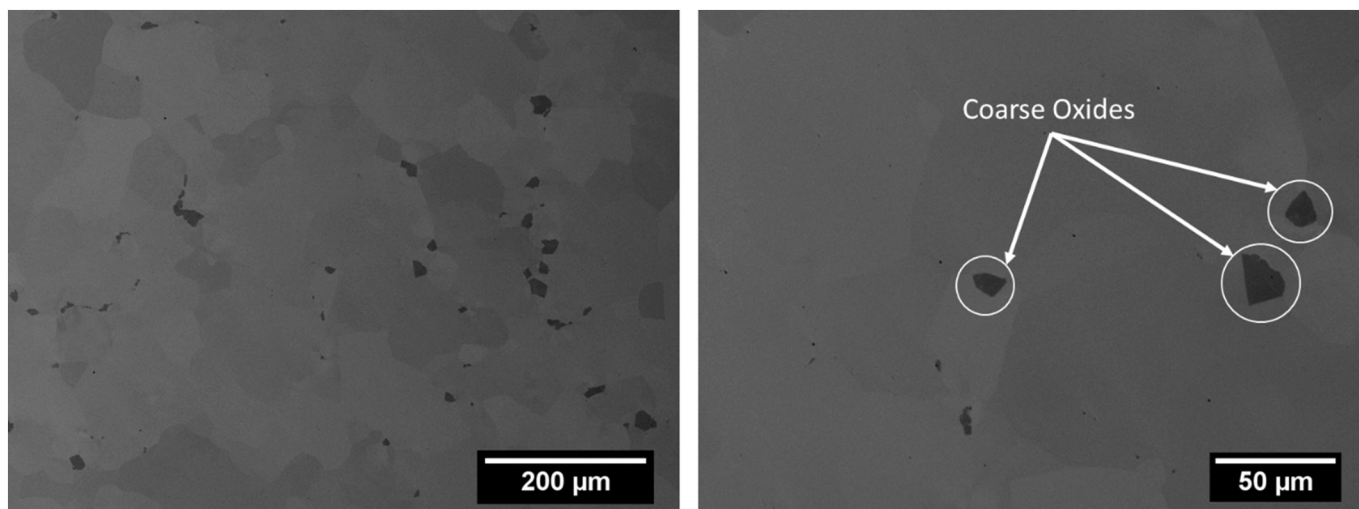


Fig. 17. Microstructure of HTed + HIPed Nb mid-range sieved powder.

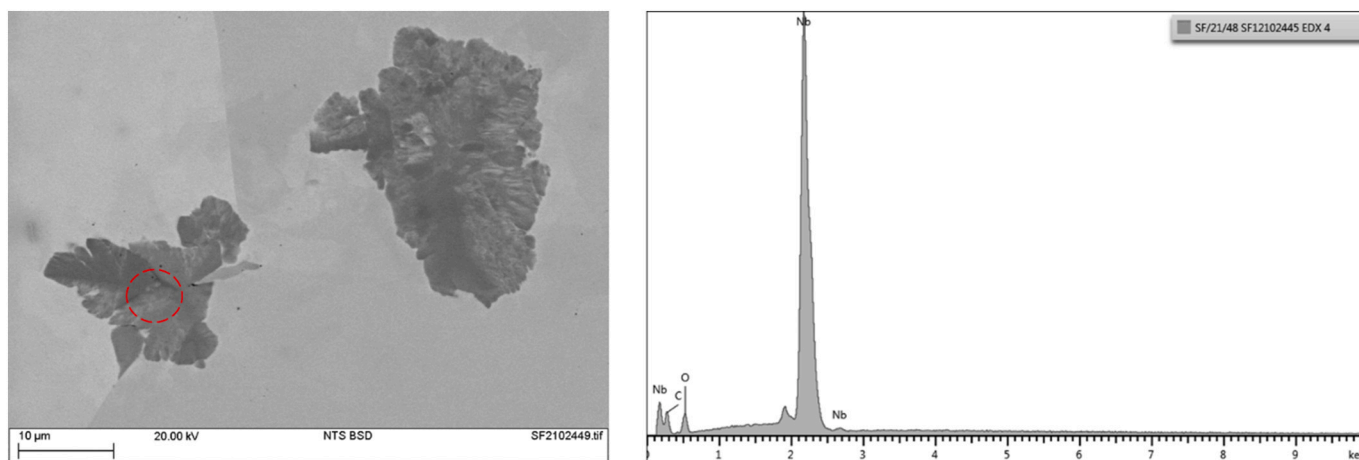


Fig. 18. EDX analysis on HTed and HIPed Nb powder.

pre-HIP heat treatment on Nb mid-range sieved powders to try to incorporate the right amount of oxygen needed to achieve the required strength.

Heat treatment of the powders was performed in air at a temperature of 300 °C for 10 h, which would guarantee enough diffusion of oxygen in the Nb powder. After heat treatment, the canister was de-gassed and HIPed using the conventional parameters used in this work.

The microstructure of the HTed + HIPed Nb powder is reported in Fig. 17. The micrographs clearly show the presence of some oxides randomly distributed in the microstructure.

The presence of oxides is confirmed by the EDX spectrum in Fig. 18 which also highlights the presence of C in the precipitates.

Some microhardness measurements were performed to understand the effect of pre-HT on the strength of the as-HIPed material. The microhardness results confirm that the pre-HT on Nb powders has successfully increased the strength of the material by increasing the hardness from 135 HV to 248 HV. In this case, the strength contribution is attributed to both solid solution strengthening and precipitation hardening due to the presence of Nb oxide precipitation.

4. Conclusions

In this work PM HIP of pure Nb was proposed as an alternative to the more complex C-103 Nb-base alloy. HIP was successfully performed on a

variety of Nb powders, showing a near-fully dense microstructure with enhanced mechanical properties. It was demonstrated that O is the main variable influencing the room temperature mechanical properties of pure Nb. It was also shown that the mechanical properties can be easily modified by changing the powders' characteristics. The microhardness of fine and coarse HIPed Nb powders showed higher values if compared to HIPed and wrought C-103.

Sieving of mid-range Nb powders has shown a significant increase in microhardness and an increase in room temperature mechanical properties while maintaining a good level of elongation. Heat treatment of Nb powders was also considered as a valid strategy, capable of increasing the O-levels over and beyond the solid solution limit, leading to oxides formation and enhanced strength.

Finally, through a simple model, it was calculated that the O-level necessary to achieve a yield strength of 260 MPa is approximately 1000 ppm.

Based on the above considerations, pure Nb processed by PM HIP can represent a valid alternative to Nb alloys for two main reasons:

- The high solid solubility of O in Nb can produce high values of yield strength.
- The flexibility of the PM HIP process allows tailoring the microstructure and mechanical properties of Nb through powder quality

and process parameters i.e. control of the oxygen levels and powder particle size.

Additionally, the study demonstrated that despite the presence of reactive elements, the as-HIPed C-103 microstructure was free from PPBs and the mechanical properties of as-HIPed C-103 were superior if compared to the minimum specifications for wrought C-103. Nevertheless, the use of solid solution strengthened Nb alloys will tendentially give a better HIP response. In particular, the addition of elements, such as W, Mo or Ta, should be considered as an addition to pure Nb for PM HIP procedure.

Despite the excellent performances of as-HIPed pure Nb, the room temperature tensile properties of wrought C-103 have not yet been achieved, thus future works should focus on optimising the room temperature mechanical properties for pure Nb. Furthermore, the high-temperature mechanical properties of pure Nb could be affected by the absence of stable precipitates hindering the grain boundary sliding. More research, therefore, is required to assess the high-temperature properties such as high-temperature tensile tests and creep.

Authors statement

Alessandro Sergi: Methodology, Investigation, Visualisation, Validation, Formal analysis, Writing – original draft. Raja H. U. Khan: Supervision, Resources, Writing – review & editing. Konstantinos Georgilas: Investigation, Formal analysis. Martina Meisnar: Resources. Advenit Makaya: Resources. Moataz M. Attallah: Conceptualisation, Supervision, Resources, Investigation, Writing – review & editing.

Declaration of Competing Interest

None.

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