

VERY HIGH-TEMPERATURE Nb-SILICIDE-BASED COMPOSITES

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Abstract

This paper describes recent progress in the development of Nb-silicide-based in-situ composites. These composites are being developed for structural applications with service temperatures of up to 1350°C. From a commercial perspective, investment casting of near netshape components of Nb-silicide composites offers substantial potential because of its proximity to existing airfoil manufacturing practices. However, investment casting techniques are not yet well developed for Nb-silicide composite airfoils. Furthermore, there is only a limited understanding of the relationship between composition, processing techniques and the properties of Nb-silicide composites. Alloying schemes have been developed to achieve an excellent balance of room temperature toughness, fatigue crack growth behavior, high temperature creep performance, and oxidation resistance over a broad range of temperatures.

This paper describes Nb-silicide-based composites with particular emphasis on processing, microstructure, and performance.

Introduction

During the past 50 years, improvements in the properties of high-temperature materials have contributed to the enhanced performance of both aircraft engines and land-based gas turbines [1]. As a result of the many nickel-based superalloy developments, advanced turbine engine airfoils now operate at temperatures up to ~1150°C. However, further increases of the temperature capability of nickel-base superalloys beyond third-generation single crystal alloys (3GSX) will be very difficult to achieve because most advanced superalloys melt at ~1350°C. Materials that can operate above the present temperature limit are required for advanced gas turbines. An increase in airfoil material temperature capability is required for several reasons, including life extension and higher combustion temperature. Turbines for either air or surface applications have a range of critical performance requirements, including fuel efficiency, thrust, weight, and reliability. The potential of Nb-silicide-based composites for high-temperature applications was first recognized approximately ten years ago [1-7]. The Nb-silicide-based composites showed early promise based on a good balance of high-temperature strength and low-temperature damage tolerance. The lower density of Nb-silicide-based systems (~7 g/cc [7-12]) was also considered a major advantage over nickel-based superalloys. It was recognized that oxidation and creep performance were areas where significant improvement was required. The progress that had been made previously in improving the oxidation performance of monolithic Nb alloys [5, 6] suggested that alloying schemes could be employed to improve the oxidation behavior of Nb-silicide composites. As a result, the effect of alloying additions on the balance of mechanical and oxidation properties has been examined in a range of Nb-silicide

composites; some of the important features of these developments will be described in this paper. There have also been several extensive reviews of recent developments of Nb-silicide based composites [1, 3, 4, 12], as well as technical papers on phase equilibria, mechanical properties, and oxidation behavior of these alloys [13-17]. Nb-silicide composites consist of Nb₃Si, and/or Nb₅Si₃ silicides, and a Nb-based metallic solid solution (Nb) with a silicide volume fraction between 0.35 and 0.6. These Nb-silicide-based DS composites are based on the metal-rich end of the binary Nb-Si phase diagram, using alloys with 12 to 25 percent Si (all compositions are provided in atom percent in this paper). In some alloys a small volume fraction (<0.15) of a NbCr₂-based Laves phase has also been used for improved oxidation resistance [1-4]. The microstructure of a simple Nb-silicide composite (Nb-15Ti-16Si) is shown in Figure 1.

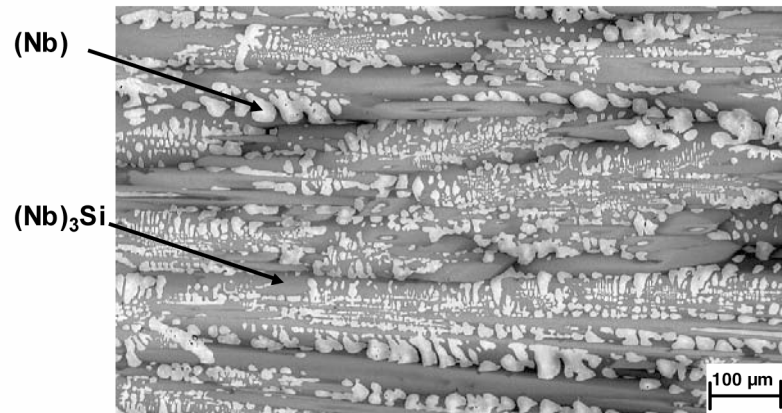


Figure 1: Microstructure (SEM backscattered electron image) of the longitudinal section of a DS Nb-silicide composite consisting of (Nb) and (Nb)₃Si. The composite is from a Nb-15Ti-16Si alloy.

This composite consists of (Nb) and Nb₃Si with a volume fraction of ~0.4 [1, 3]. The Nb₃Si with Ti in solid solution is generally referred to as (Nb)₃Si. The Nb-silicide can exist in three different crystal forms: (Nb)₃Si - (tP32), (Nb)₅Si₃ - (tI32), and (Nb)₅Si₃ - (hP16). A full set of properties was described for an early Nb-silicide-based composite, MASC, which had a composition of Nb-25Ti-8Hf-2Cr-2Al-16Si [10]. At present, there is only limited understanding of the relationships between composition, processing technique, and properties, but previous alloying efforts have shown some compositional trends for optimizing properties including oxidation resistance, toughness and creep [1, 3-7, 12]. Recent compositions have been developed at GE; in this paper, they have been designated Nb-silicide A, B [16, 17], C [1, 16], and D [18]. Nb-silicide A has a composition of Nb-25Ti-8Hf-16Si. The compositions of Nb-silicide C and D fall within the ranges described in the referenced patent [17].

Nb-silicide-based composites have been fabricated using a range of processes, including nonconsumable arc-melting, plasma arc melting, induction skull melting, ingot casting plus thermomechanical processing [4, 11, 13, 15], directional solidification (DS) [1, 3], investment casting [7], and powder-metallurgy processing [1, 3]. From a commercial point of view, investment casting of near-net-shape components of Nb-silicide composites offers substantial potential because of its proximity to existing complex airfoil manufacturing practices. However, investment casting techniques are not well developed for Nb-silicide composite airfoils. In addition, the reactivity of molten Nb alloys limits the use of ceramic-based melting systems. Recently, investment casting of Nb-silicide composites has been explored [5]. A typical prototype airfoil is shown in Figure 2.

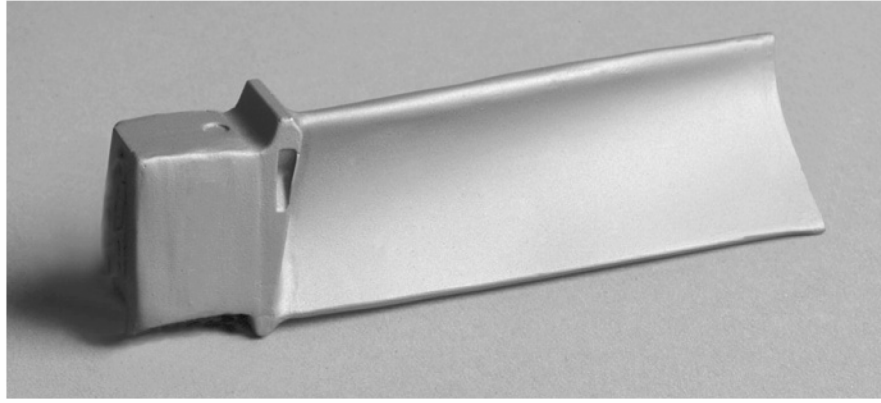


Figure 2: Typical Nb-silicide composite prototype airfoil produced by investment-casting; the airfoil is approximately 150mm long.

The aim of this paper is to describe the most important features of recent Nb-silicide composite developments. In the following sections, mechanical properties and oxidation performance, including coatings behavior, are described.

Mechanical Properties

Fracture and Fatigue Performance

There has been a significant amount of basic research on fracture and fatigue performance of Nb-silicide-based composites based on binary Nb-Si and higher order systems [19-25]. Figure 3 shows typical room temperature fracture toughness measurements, and a typical fracture resistance, or R-curve, for the Nb-silicide C.

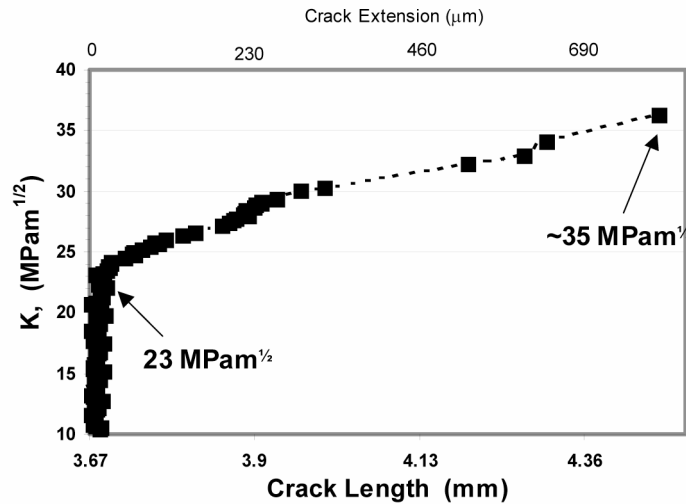


Figure 3: Typical R-curve of a DS Nb-silicide-based composite. For this sample, the R-curve shows an initiation toughness of 23 MPa m^{1/2} and a peak toughness of ~35 MPa m^{1/2}.

The R-curve shows an initiation toughness of 23 MPa m^{1/2}, and a peak toughness of ~35 MPa m^{1/2}. A range of initiation toughness values between 15 and 28 MPa m^{1/2} was measured for different samples of Nb-silicide C. Similar toughness levels have been reported in other

Nbsilicide-based systems [12-16], and in similar composites made by other methods, such as investment casting and extrusion; these are very high toughness levels for an intermetallic-based system. R-curve behavior is generally observed in Nb-silicide-based composites [3]. Higher strain rate toughness testing has also been performed and it was found that there is little sensitivity of peak toughness to loading rate [19]. This is consistent with the behavior of polycrystalline Nb, which has been tested over a wide range of temperatures and loading rates [19, 23-26]. Fatigue tests performed at room temperature showed DK thresholds of 7-11 MPam^{1/2} and Paris law slopes of 7-12 for Nb-silicide C. Fatigue tests were performed over a range of DK values of 9 to 20 MPam^{1/2} with an R value of 0.1, as shown in Figure 4.

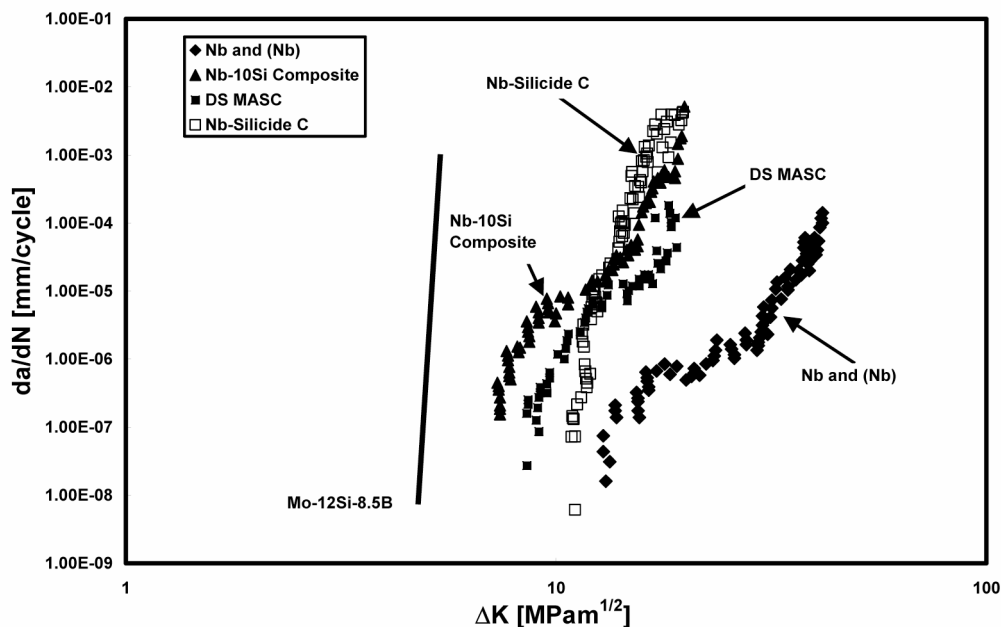


Figure 4: Fatigue data for three Nb-silicide composites: Nb-silicide C (open squares), MASC (solid squares), and a Nb-10Si composite (solid triangles). Data for a series of monolithic Nb alloys (pure Nb, Nb-1Si, Nb-1Zr) (solid diamonds) are also shown [23-26]. Literature data for a Mo-Si-B alloy are also included for comparison [19, 20].

For these particular fatigue tests, the Paris law slopes were in the range of 7-12. In comparison, the Paris law slope for a typical airfoil nickel-based superalloy is ~5. Figure 4 provides a comparison of the fatigue data for the Nb-silicide C with fatigue data for monolithic Nb solid solutions [23-26], the MASC composite, and a Nb-10Si Nb-silicide in-situ composite [19]. The data show that the fatigue behaviors of a range of Nb-silicide-based composites are approximately metallic-type, and the Paris law slopes of the Nb-silicide-based composites are only slightly higher than those of the monolithic Nb solid solutions. Literature data for a Mo Si-B alloy are also included for comparison [19, 20].

Tensile and Compression Behavior

The strength of a range of Nb-silicide-based composites is shown in Figure 5 for temperatures from room temperature to 1350°C (2460F). UTS data for a typical second generation single crystal nickel-based superalloy are also shown. Room temperature tensile strengths of ~830MPa were reported in early Nb-silicide-based composites with Cr concentrations of ~2% [10]. The more recent Nb-silicide C displayed compression strengths as high as ~1700 MPa at room temperature, 520MPa at 1200°C, and 310MPa at 1350°C. The compression strength level

of ~1700 MPa of the Nb-silicide C was almost constant from room temperature to 800°C. At 1200°C, the compression strength of Nb-silicide C is more than twice that of the tensile strength of a typical second generation single crystal nickel-based superalloy. Some of the more recent Nb-silicide composites have high room temperature compression strengths, but lower tensile strengths. Pores and defects can lead to a reduction in the maximum tensile strengths. Refinement of silicide phases by innovative processing (including powder metallurgy) can reduce the defect size and increase the tensile strength, while maintaining adequate creep performance.

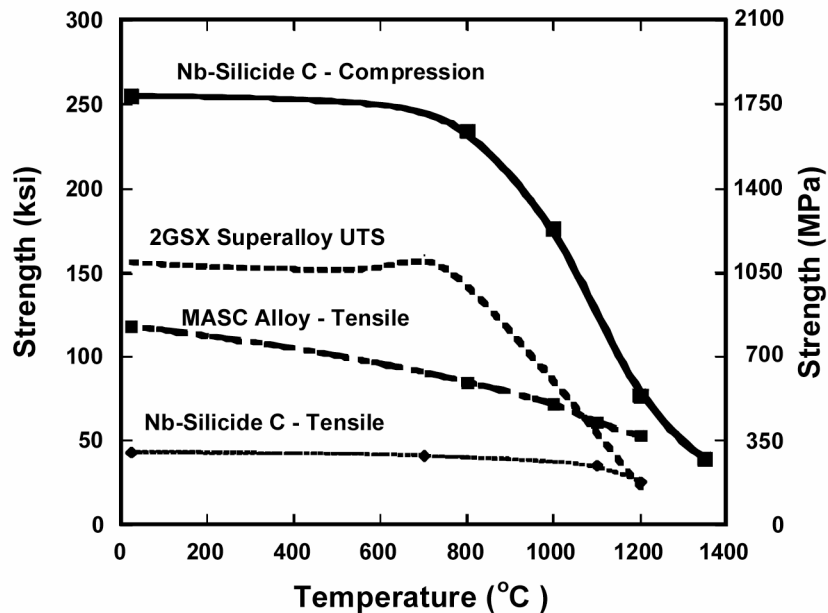


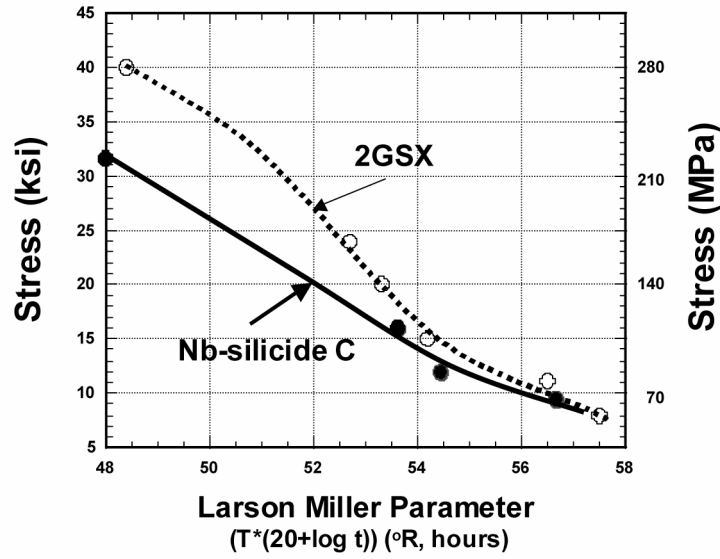
Figure 5: Comparison of the strength of Nb-silicide-based composites for temperatures from room temperature to 1350°C. The strength of DS Nb-silicide-based composites is shown in both compression and tension. Tensile data for a typical second generation single crystal nickel-based superalloy are also included.

High temperature extrusion has been performed to examine the effect of microstructural scale on the tensile properties for higher Cr concentration alloys [1]. Compression, tension and toughness tests have been performed from room temperature to 1200°C for cast alloys, and up to 1100°C for the extruded alloys [1]. The compressive yield strengths were high at room temperature and 800°C (1550-1600 MPa and ~1500 MPa, respectively), and they decreased to between 500-600 MPa at 1000°C. Typically the ductility values in these composites are ~3% in compression at room temperature, and higher than 3% at temperature above 800°C. These materials can exhibit a brittle-to-ductile type transition at temperatures >1000°C, based on macroscopic ductility measurements.

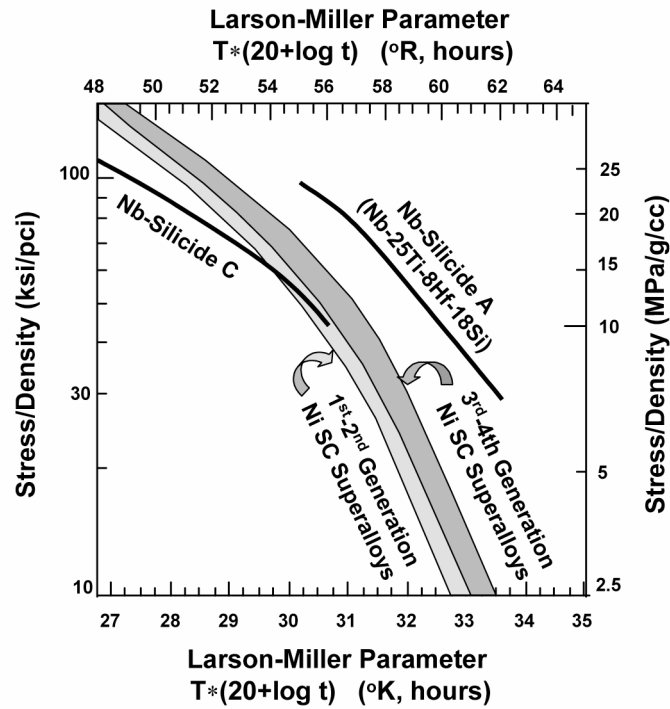
Creep Performance

Figure 6 shows both the tensile creep stress (Figure 6a) and the density-normalized creep stress (Figure 6b) as a function of Larson-Miller parameter using the time to rupture for the DS Nb silicide C. Data for typical second and third generation single crystal nickel-based superalloys are also shown. The performance of Nb-silicide C is similar to the second generation single crystal nickel-based superalloy at stresses up to 100MPa. The Nb-silicide C composite shows improved density-normalized creep performance over the superalloy at low stresses, and

slightly inferior performance at high stresses. Similar comparisons are obtained when using the time to 1% strain for the Larson-Miller parameter ($^{\circ}\text{R}$, hours, and K hours), rather than the time to rupture [3].



(a)



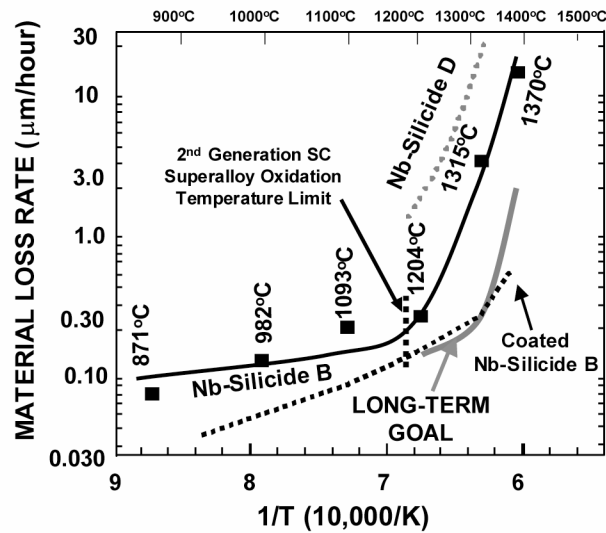
(b)

Figure 6: Creep stress (a) and density-normalized creep stress (b) as a function of Larson-Miller parameter for the Nb-silicide C composite. Data for typical second generation (2GSX) and third generation (3GSX) single crystal nickel-based superalloys are shown for comparison. Considering the creep-stress, the performance of the DS Nb-silicide-based composite is similar to the second generation single crystal nickel-based superalloy at stresses up to 100MPa.

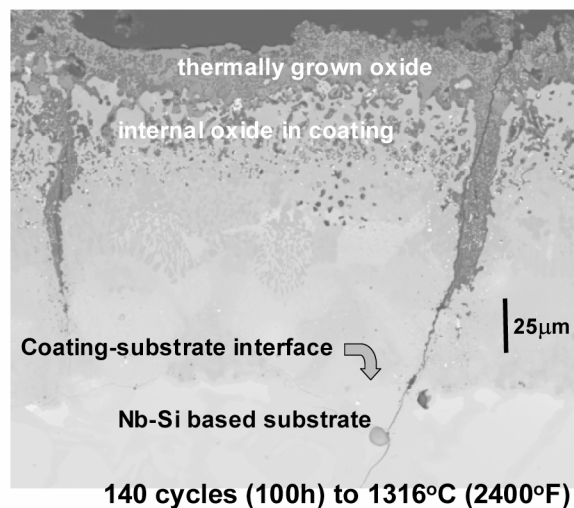
The density-normalized creep rupture behavior of the Nb-silicide A composite (Nb-25Ti-8Hf-18Si) is also shown in Figure 6 [7, 12]. The Nb-silicide A shows an improvement of ~ 2 Larson-Miller parameters over present single crystal nickel-based superalloys [27]. The creep rupture

behavior of the Nb-silicide C is similar to that of the second generation single crystal superalloys (2GSX). An increase in specific rupture performance results from the lower density of the Nb-silicide in comparison with the 2GSX superalloy.

For an airfoil alloy, the goal for creep performance is that there should be less than 1% creep in 125 h at temperatures of 1200°C and stresses of >170 MPa. There is generally minimal primary creep in Nb-silicide composites; this corresponds to a secondary creep rate of $2.2 \times 10^{-8} \text{ s}^{-1}$. The secondary creep behavior of a range of Nb-silicide-based composites has been described previously [3-6]; creep rates of $< 3 \times 10^{-8} \text{ s}^{-1}$ at 1200°C and stresses greater than 170 MPa have been demonstrated [3, 28]. At temperatures less than 1000°C, it is difficult to measure the creep rates of Nb-silicide composites since they are $< 10^{-9} \text{ s}^{-1}$. Creep of Nb-silicide-based composites is controlled by a combination of creep in both the silicide and metallic phases [3, 5]. Monolithic phase studies have also indicated that complex tI32 Nb⁵Si³ type silicides offer improved creep performance in comparison with complex tP32 Nb³Si type silicides [3]. The secondary creep of monolithic Nb⁵Si³ essentially defines the lowest secondary creep rates that can be expected in these composites.



(a)



(b)

Figure 7: (a) Cyclic oxidation data (measured material loss) for Nb-silicide composites as a function of temperature, showing the order of magnitude increase in oxidation resistance of the most recent Nb-silicides, Nb-silicide B, in comparison with those developed in 1998, Nb-silicide D [5, 18].

Figure 7b shows a micrograph of a coated Nb-silicide composite oxidized at 1316°C for 100 hours.

Composite Oxidation Performance

Figure 7 shows the typical cyclic oxidation performance for Nb-silicide-based composites as a function of temperature. Figure 7 shows the increase in oxidation resistance of the recent Nbsilicides (~2002) in comparison with those developed prior to about 1998 [3]. The current best Nb-silicide-based composites possess a temperature capability ($<25\text{ }\mu\text{m}$ lost in 100 hours) of $\sim 1200^{\circ}\text{C}$. Both short term and long term cyclic oxidation resistance goals have been established for Nb-silicide composites. The short-term goal corresponds to a loss of $<200\text{ }\mu\text{m}$ in 10 hours of test stand exposure at 1370°C (for test). The long-term goal corresponds to a loss of $<25\text{ }\mu\text{m}$ in 100 hours at 1315°C (for service). The basis for these oxidation goals are the requirement to achieve the same oxidation life at 1315°C that 2GSX superalloys presently display at 1150°C [1, 3, 12, 27]. The present uncoated Nb-silicide composites satisfy the short term goal because they lose $<200\text{ }\mu\text{m}$ in 10 hours of test stand exposure at 1370°C . However, the long-term goal requires a further order-of-magnitude reduction in material loss upon oxidation. The oxidation performance of coated Nb-silicide composite meets the long-term uncoated alloy goal.

Some refractory metals can also experience pesting damage at intermediate temperatures (generally $<850^{\circ}\text{C}$). Pesting involves preferential oxidation at microstructural features, such as grain boundaries; it can lead to self-pulverization in samples cycled from room temperature to relatively low temperatures and it is generated by oxygen embrittlement. Alloying Nb-silicide composites with tin has been shown to be very effective in controlling pesting damage, in that such damage has been essentially eliminated in the temperature regime of 750°C to 950°C [7]. Present Ni-based superalloy airfoils are designed with bond coats and thermal barrier coatings (TBCs) so they can withstand very high airfoil surface temperatures. There have also been significant developments of bond coats and thermal barrier coatings for Nb-silicide-based composites [7]. The high performance oxidation-resistant coatings can provide protection for

Nb-silicide-based composites at temperatures of $>1370^{\circ}\text{C}$ for more than 100 hours (Figure 7a). A typical silicide coating is shown in cross-section in Figure 7(b) after oxidation at 1316°C for 100 hours (140 cycles). There was less than $25\text{ }\mu\text{m}$ of coating loss. Cracks that formed in the as deposited coating rapidly oxidized and were effectively blunted and sealed, minimizing oxygen penetration to the substrate. The cracks did not propagate with further high-temperature exposure or thermal cycling. A layer of thermally grown oxide (TGO) formed and there was some internal oxidation in the outer region of the coating, indicating that the coating still had significant life remaining beyond the 100 hour exposure. There was little coating-substrate interaction, reflecting the excellent stability of the silicide coating. Significant substrate internal oxidation occurred in the uncoated condition, but virtually no internal oxidation in the coated condition.

A series of aggressive tests, including furnace cycling tests (FCT) and controlled temperature gradient, jet engine thermal simulation tests (JETS), have been performed on TBC-coated Nbsilicide composites. The Nb-silicide substrates coated with the bond coat-TBC system were JETS tested as shown in Figure 8. Coupons were subjected to a high thermal gradient by heating the front face of the TBC with a high-velocity combustion flame generating a TBC temperature of 1650°C and cooling the rear surface with a cooling air nozzle so that the Nbsilicide had a temperature of $>1200^{\circ}\text{C}$.

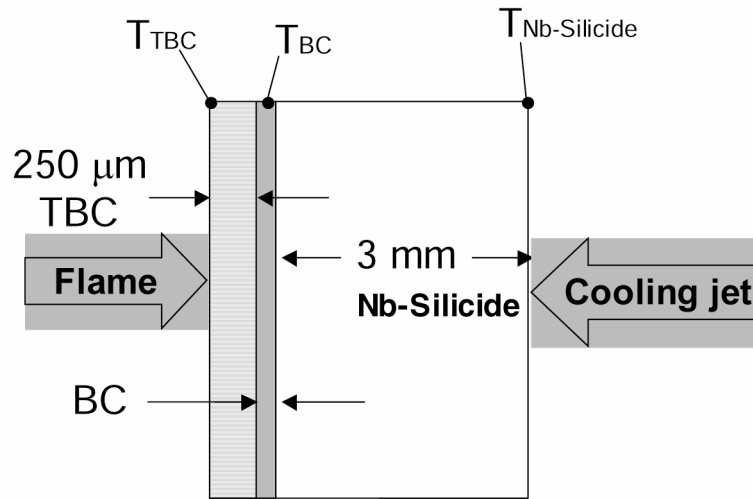


Figure 8: Schematic diagram showing the configuration used for the jet engine thermal simulation testing (JETS) of Nb-silicide composites. The diagram shows a cross-section of the sample (including the bond coat and the substrate) together with the hot flame and the cooling jet. Each test cycle consisted of exposure to the thermal gradient for 1 hour followed by a 15 minute cool [7].

The coating was tested cyclically in FCT at both 1370°C (2400°F) for 100 hours and 1430°C (2600°F) for 50 hours, with no damage to the TBC or bond coat, and no substrate internal oxidation. In JETS testing, coated samples survived >10 cycles with a TBC temperature of 1370°C, with an additional 10 cycles to 1510°C (2750°F) and a further six cycles to 1650°C (3002°F) before TBC delamination occurred in cycling to 1790°C (3250°F). For a sample removed after 1650°C, essentially no damage of the TBC and bond coat detected. The performance of the coated Nb-silicide composite in both the FCT and JETS tests is very promising.

Summary

Nb-silicide composites offer excellent potential for high-temperature applications. This article has focused on using Nb-silicide composites in turbine engine applications. Nickel-base superalloy airfoils presently have maximum operating surface temperatures of ~1150°C, and future designs require materials that can operate at temperatures above 1300°C. Recent Nbsilicide-based composites have shown excellent progress in developing processing techniques, creep rupture resistance, fracture and fatigue crack growth performance, oxidation resistance, and coatings. Nb-silicide composites offer potential temperature capability of >1100°C together with ~20% lower density than 2GSX superalloys.

Recent investment casting mold developments have shown excellent potential for thin wall airfoil casting. The oxidation resistance and creep performance of present Nb-silicide composites is satisfactory for near-term engine testing, but further improvements are required to satisfy the long-term design requirements. In addition, the unique bond coats for Nb silicidebased in-situ composites have demonstrated excellent oxidation resistance at temperatures up to 1370°C. Thermal barrier coatings have also been explored, and thermal cyclic oxidation testing has shown excellent retention/survivability of the TBC at temperatures

of up to 1650°C in a JETS test.

Key technical challenges are in the areas of processing, oxidation resistance, and creep rupture resistance. Manufacturing complex shaped Nb-silicide components poses significant challenges and substantial process developments are required to manufacture complex articles. However, investment casting of Nb-silicide-based composites offers a relatively low-cost processing approach for near net-shape components.

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