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High temperature compression strength and oxidation of a V-9Si-13B alloy

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ABSTRACT

Vanadium-based alloys are attractive relative to nickel base superalloys for high temperature applications due to significantly reduced density and increased melting temperatures. In this exploratory study, the compressive strength and oxidation behavior of a powder metallurgically processed V-Si-B multiphase alloy is examined. Results are compared to CMSX-4 and other existing vanadium alloys. Compressive yield strength is comparable to the nickel base superalloy at 1000 °C but the mass gain during isothermal oxidation at 600 °C is higher, implying the need for a protective coating for sustained high temperature exposure.

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Materials attributes such as high-temperature strength and creep resistance, which typically scale with the melting point, dominate the development and materials selection strategies for high temperature applications. Several new alloys and compounds based on elements with ultra-high melting points like molybdenum, niobium and platinum are currently being investigated/developed [1-4]. Moreover, the weight of the components, particularly for accelerating or rotating structural components is a major consideration and accordingly, alloy density is a key factor in materials selection. In this regard, vanadium is a promising material for specific structural applications as it provides the lowest density of the various high melting point metals; therefore, vanadium has much higher density-normalized ultimate tensile strength than nickel up to elevated temperatures [5].

Some low-alloyed vanadium-based materials are the focus of current research: for example, alloy V-4Cr-4Ti was identified as an attractive material for self-cooled liquid Li blanket in fusion reactors [6]. Nevertheless, pure and low-alloyed vanadium have poor oxidation resistance [7,8]. To improve the oxidation behavior, it was found that Ti, Cr, Al, Si and B additions help to reduce the mass change at temperatures between 600 °C and 800 °C [7,8]. Furthermore, such low-alloyed vanadium materials demonstrate a drop in strength above temperatures of 600 °C [9]. Nanoparticle strengthening by Y₂O₃ and TiC leads to an improvement in yield strength by 25 MPa-180 MPa at 700 °C compared to the base alloy V-4Cr-4Ti and providing yield strength values about 415 MPa [10].

To enhance the strength at high temperatures and thereby extend the performance envelope, strong and creep-resistant phases need to and was not concerned with evaluating the properties of the consolidated powders in such binary compositions. This study focuses on the behavior of a ternary V-Si-B three-phase alloy, produced using a powder metallurgical processing route. To enhance high temperature strength, this alloy includes the two intermetallic phases, V₃Si and V₅SiB₂, in addition to the vanadium solid solution phase. An alloy of composition V-9Si-13B (in at.%) was chosen from the ter-

be incorporated in a vanadium solid solution matrix in sufficient quan-

tities to generate a "composite". For example, such composite materials

could include ultra-high strength intermetallic phases such as V₅Al₈,

V₃Si, V₅Si₃ or V₅SiB₂ [11–14]. Of these, the Si and B containing phases

have the potential to improve the oxidation resistance significantly

[8]. However, the synthesis of such materials is difficult because of the

extremely high melting temperatures of the components. Bei et al. [14] produced V-V₃Si eutectic composites using a directional solidifica-

tion method, which resulted in a well-defined lamellar microstructure

and enhanced mechanical properties compared to the vanadium solid

solution alloy V-4Cr-4Ti. Nevertheless, this process requires knowledge

of the eutectic composition in the binary V-Si system and also cannot be

applied on other, non-eutectic, binary or ternary alloy compositions. Liu

and Ciu [15] used mechanical alloying to process powders of different

binary mixtures of V-Si and they investigated the mechanisms

governing solid-state reactions yielding V₅Si₃ and V₃Si phases. Howev-

er, their work focused on the evolution of the powder microstructure

nary phase diagram presented by Nunes [12]. The intention of selecting this composition was to control the proportion of intermetallic phases and the solid solution phase. This would enable a balance in high temperature strength and low temperature ductility. This alloy was powder metallurgically processed from elemental powders of V, Si and B with a purity of 99.9, 99.8 and 98%, respectively. The powders were handled





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Fig. 1. a) X-ray diffraction data for the mechanically alloyed and the annealed powders of the V-9Si-13B alloy confirming the formation of a three-phase material from a V(Si, B) solid solution; microstructure of b) an annealed powder particle and c) compacted V-9Si-13B.

and milled under a protective argon atmosphere in a Retsch® PM400 planetary ball mill for 25 h at 200 rpm to form supersaturated V(Si, B) solid solution powders. All milling conditions as well the hardening effects in the solid solution phase are described in more detail in [16]. It is noted that the milling characteristics of binary V-Si [16] are comparable to ternary V-Si-B mixtures since the effect of low boron concentrations on the milling behavior is negligible, a characteristic that was previously verified for Mo-Si and Mo-Si-B powders [17]. Next, the milled V-9Si-13B powders were used to study the precipitation response of the supersaturated V(Si, B) solid solution by conducting annealing experiments at 1400 °C, 1500 °C and 1600 °C in an Ar atmosphere with the intention to identify an appropriate temperature for the subsequent compaction process. X-ray diffraction (XRD) measurements were performed using an X'Pert Powder X-ray diffractometer (PANalytical) using Cu K_{α} radiation. The phase identification was performed using the analysis software X'Pert HighScore Plus (PANalytical).

After mechanical alloying, the V-9Si-13B powders were compacted using the field assisted sintering process at 1500 °C following the parameters used for Mo-Si-B alloys in a previous study [18]; this resulted in a material with a density of 5.85 g/cm³ (measured by the Archimedes' method). This is marginally higher than the theoretical density of 5.77 g/cm³, calculated using Vegards law. The binary phase V-Si, V-B and B-Si diagrams, the isothermal section of the ternary V-Si-B phase diagram [12,19,20] and the densities of V₃Si, V₅SiB₂ and the V solid solution phase were used for input data [13,21]. The difference in the measured and calculated density are attributed to inaccuracies of the phase diagrams in the literature, and the assumption of phase stability below 1600 °C due to the lack of information on the room temperature ternary isotherm for the V-Si-B system.

The microstructure of the V-Si-B material was investigated using scanning electron microscopy (SEM) in the back scattered electron mode (BSE) combined with Energy Dispersive X-ray (EDX) analysis. Samples for SEM investigation were prepared from the compact buttons by electro-discharge machining and grinding from 180 grit down to 1200 grit, followed by mechanical polishing with a 3 μ m and 1 μ m diamond suspension successively and then finished using colloidal silica. Even though backscattered electron imaging differentiates the phases present in shades of grey, automated image analyses could not successfully separate V₃Si from V₅SiB₂. To overcome this problem, the program Image J was used to mark the two phases in the SEM micrographs manually for the determination of the individual phase fractions subsequently using image analysis program.

The phases present in the consolidated samples were identified by X-ray diffraction using Cu K_{α} radiation. Microindentation tests were performed using a Vickers indenter at a load of 0.1 N and a hold period of 5 s. The microhardness measurements were performed by generating a series of indents at a predetermined load at well-defined intervals. SEM backscattered electron imaging was then used to assign the microhardness data to the individual phases that were distinguished based on atomic number contrast (different grey values); further only those data were included where the indent was fully within the individual particle. The mechanical behavior was evaluated using constant displacement tests at a nominal strain rate of $1 \cdot 10^{-4} \text{ s}^{-1}$ in uniaxial compression at temperatures between 600 °C and 1100 °C in a flowing Ar/H₂ atmosphere using a Zwick/Roell Z100 electro-mechanical testing machine equipped with a Maytec furnace. The yield stresses were measured by the 0.2% offset method. Compression samples with a cross-section of $3 \times 3 \text{ mm}^2$ and a height of 5 mm were prepared from the V-Si-B compact by electro-discharge machining, grinding down to 1200 grit and mechanical polishing with a 3 µm diamond suspension. For purpose of



Fig. 2. Microhardness of the individual phases in the V-9Si-13B alloy compact.

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