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ANALYSIS OF AN ALLOYING PROCESS FOR NBSI-BASED COMPOSITES IN A COLD CRUCIBLE INDUCTION FURNACE WITH THE USE OF THE PARTICLE TRACING METHOD

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Refractory metals based on niobium are progressively attracting interest in the industrial world for the production of components used in high-temperature environments. With superior thermal and mechanical properties compared to those of nickel-based superalloys, NbSi composites represent a promising alterative for the fabrication of turbine components, and they open a way to the further increase of their efficiency. But the melting process of such refractories still faces relevant technical limitations related to their high melting point, chemical reactivity and poor homogenization of the alloy. For these reasons, numerical simulations play a fundamental role in detailed analysis of their melting process. With the help of multiphysical simulations and the particle tracing method, the addition of targeted metals into the NbSi base is investigated and optimized.

Introduction. The selection of appropriate material properties plays a key role in the fabrication of components used in high-temperature environments. A significant example is the blades installed in the high-pressure stadium of turbines for either air or surface applications. Such high-performance components are permanently exposed to very harsh operating conditions, like enormous mechanical stresses, vibrations and extreme temperatures, typically higher than 1100°C: these may result in fatigue failure and limit the longevity of the blades. The modern fabrication of turbine blades deals mainly with nickel-based superalloys, which stand out mostly for their excellent creep behavior and corrosion resistance. Yet, these alloys are characterized by major throwbacks like high density in the range of $8.15-8.35\,\mathrm{g/cm^3}$ and limited operating temperatures of maximum 1150°C [1]. Further increases in their temperature capability will be very difficult to achieve, since the most advanced superalloys melt at 1350°C.

The need for future improvements of the turbines, especially in terms of energy efficiency, is driven by the increase of the permissible operating temperature of their materials. In this regard, innovations in refractory alloys are being pursued, with particular interest in composites based on niobium (Nb) and silicon (Si). The systems named niobium-MASC (Metal and Silicide Composites) are characterized by a high melting point over 1750°C and have the potential for service at temperatures higher than 1350°C. With a density of 6.6 to $7.2\,\mathrm{g/cm^3}$, lighter turbine blades can be fabricated and, for conventional aircraft applications, this translates into a lower fuel consumption and minimization of CO_2 emissions [2]. The phases of niobium and silicon complement each other, providing good fracture toughness, high-temperature strength and oxidation resistance (Fig. 1). The targeted addition of other elements – aluminium, titanium, chromium and hafnium – improves complementary properties, such as the creep resistance and fracture toughness. In contrast to these advantages, the controlled production of such alloys faces

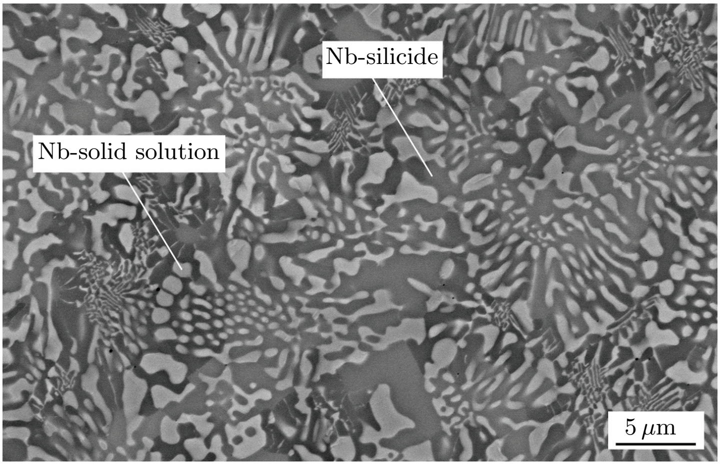

Fig. 1. Microstructure of the $Nb_{18}Si$ alloy. Source: Institut für Werkstoffkunde (IW) of the Leibniz University Hanover.

Fig. 2. The induction crucible furnace with cold walls. Source: Institute of Electrotechnology (ETP) of the Leibniz University Hanover.

major technical challenges: chemical reactivity of niobium melts essentially excludes the use of ceramic-based melting systems because the mutual contamination of the melt and crucible can affect the quality of the final cast. Furthermore, the longevity of the crucible is compromised by extreme thermal loads.

The successful fabrication of Nb-silicide-based composites by alternative processes is reported in few works: the cited technologies include arc melting, ingot casting plus thermomechanical processing, directional solidification, vapor deposition and powder-metallurgy processing [3–6]. Despite the successful production of laboratory-scale volumes of the alloy, the investment of energy, materials and time remains significant for all these techniques. Moreover, the good quality of the product is not guaranteed. In this work, semi-levitation melting by electromagnetic induction in a cold crucible furnace (ICCF) is proposed as an innovative approach to process melts based on niobium and silicon: due to the generation of strong electromagnetic forces inside the workpiece, this technology enables largely contactless melting, which prevents contamination, ensures higher purity of the workpiece and hence minimizes the costs related to the crucible materials (Fig. 2). The melt is affected by strong electromagnetic stirring that makes the ICCF an excellent solution for mixing and alloying multi-component systems. Strong homogenization is expected, with a significant improvement of the properties of the solidified cast. Melting by electromagnetic induction in the ICCF was intensively investigated by the authors for metals and alloys with a wide range of melting temperatures [7–9], but only in the recent years it was applied to niobium-based materials: a sample of pure niobium with a volume of 100 cm^3 was melted under the supplied active power 113 kW, at the frequency 10.6 kHz. A binary alloy of niobium and silicon was successfully melted from the respective atomic masses of 82% and 18%. Very stable and reproducible melting conditions were reported even for different volumes of the sample, in particular, a stable meniscus of the melt, the working frequency 10.5 kHz and the supplied power 50 kW [10]. Starting from those preliminary investigations, this work aims to report the advances achieved by the authors in the production of Nb-silicide-based composites: the obtained matrix of niobium and silicon (NbSi) is enriched with "additional metals" – aluminium (Al), hafnium (Hf), chromium (Cr) and titanium (Ti), expecting a substantial modification of the microstructure of the alloyed product. However, the set of dissimilar material properties still imposes challenging limits to the stirring process and affects the homogenization of metals within the matrix. In this regard, numerical models take a central role in the estimation of the operational parameters of the melting process, and they are proposed to analyse the distribution of these additional elements within the NbSi base. The dispersion of metals, before their complete alloying, is calculated with the help of the Discrete Phase Model, and their motion inside the continuum is predicted with the particle tracing method.

1. Numerical model and governing equations.

The melting process by electromagnetic induction takes place in a laboratory-scale ICCF made of copper. The walls are cooled by water to withstand the severe melting temperatures and provide the generation of a protective skull for the melt. A copper inductor surrounds the crucible in symmetrical position to it (Fig. 3), and the entire

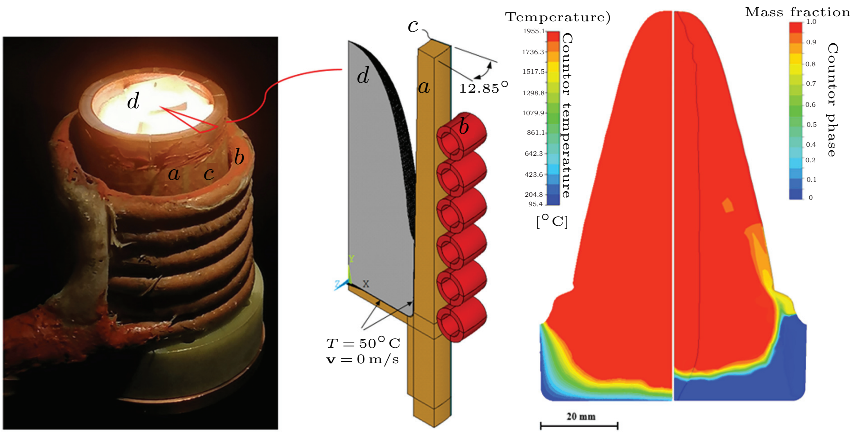

Fig. 3. The experimental setup (left), its corresponding numerical model with half palisade (center), and computational results of the induction melting process for $Nb_{18}Si$ (right). (*a*) Palisade of the crucible, (*b*) inductor, (*c*) insulating layer, and (*d*) melt. The simulation results show the thermal field and the solid/liquid fraction achieved inside the melt during the process.

setup is installed inside a protective chamber, where either a vacuum atmosphere is generated or an inert gas can be injected if necessary. Electric power is supplied to the inductor using a frequency converter produced by the EMA-TEC Company and installed at the Institute of Electrotechnology: the converter provides a maximum active power of 300 kW in the frequency range of 8 – 12 kHz.

A computational analysis of the melting and alloying process is carried out with the commercial software ANSYS. The geometry of the 3D numerical model is made of an azimuthal segment of the crucible modelled with symmetry planes: the segment is equivalent to the angle 12.85 degrees in the azimuthal coordinate and the conditions of symmetry are defined at the sides. The model comprises 1/2 crucible palisade, one insulating layer and the related arc of the inductor. Assuming a symmetrical deformation of the free surface of the melt, its corresponding arc fraction is modelled, too. Such geometrical simplification of the numerical setup is allowed by the crucible symmetry, and it is aimed at minimizing the computational cost of the simulation. If compared to the full 3D geometry, the decrease of the resulting quality is expected to be irrelevant.

The multiphysical model couples three different fields: electromagnetic (EM), fluid dynamic (FD) and thermal (TH), with the use of the Mechanical APDL and Fluent packages. The calculation of the melt free surface dynamics has been intensively investigated by multiple authors, and relevant works have been published in this field [7, 12, 13]: the EM step calculates the distribution of electromagnetic forces and Joule heat losses within the workpiece in a harmonic and steady-state regime; EM results are then imported to ANSYS Fluent, where the free surface deformation of the liquid workpiece is first computed using the Volume of Fluid method. Flow and thermal fields are calculated in parallel, as the distribution of the Lorentz forces and Joule heat is cyclically computed on the base of melt deformation. A transient regime is applied to FD and TH simulations. A typical Reynolds number for liquid niobium and NbSi-based composites in the investigated ICCF indicates a fully developed turbulent flow:

$$\mathrm{Re}_{\mathrm{Nb}} = \frac{r_0 u \rho}{\mu} \approx 10^5, \tag{1}$$

where r_0 is the characteristic length of the crucible, u is the flow velocity, ρ is the mass density of the melt, and μ is its dynamic viscosity. For this reason, the LES turbulence model is chosen for the fluid dynamic calculation. The thermal field within the workpiece is computed with the Energy and Solidification/Melting models, where the liquid fraction β is defined as

$$\beta = \frac{T - T_{\mathrm{solidus}}}{T_{\mathrm{liquidus}} - T_{\mathrm{solidus}}} \quad \text{if} \quad T_{\mathrm{solidus}} < T < T_{\mathrm{liquidus}}, \tag{2}$$

or $\beta = 0$ and $\beta = 1$ if the temperature of the body is, respectively, lower and higher than the *solidus* temperature and the *liquidus* temperature. The boundary conditions applied to the FD and TH model include: (*a*) zero velocity ($\mathbf{u} = 0$ m/s of the liquid flow on the contact surfaces between the workpiece and the crucible (bottom and side); (*b*) free velocity evolution on the free surface of the melt, with the definition of zero shear stress; (*c*) a constant temperature of $T = 50°$C imposed at the contact surface between the crucible (side and bottom) and the workpiece. This simulates a fictitious water cooling effect as it is not directly modelled. Additional numerical conditions include thermal losses by radiation with a constant emissivity of $\varepsilon = 0.36$, and thermal convection is neglected.

1.1. Use of particle tracing to investige the alloying process. The particle tracing method was intensively applied by Kirpo and Ščepanskis to investigate the motion of solid inclusions in liquid metals [14, 15]: the motion of oxide particles under the effect of electromagnetic induction was analysed with the aim to identify the regions of the melt, where undesired inclusions could agglomerate in crucible furnaces. Oxide particles were modelled as discrete phases and remained separated from the melt. This study proposes particle tracing as a tool to predict the distribution of additional metals (Al, Ti, Cr and Hf) in the NbSi base. The elements are defined as spherical solid particles moving inside the NbSi melt; this is named *continuum* in fluid dynamics. Due to the nature of the Discrete Phase Model (DPM), the simulation is limited to the mixing process prior to the complete alloying, and the particles interact with the *continuum* in terms of *momentum* and heat exchange only. The motion of particles is affected by the buoyancy, gravity forces, Stokes drag force and the EM force; no Saffmann's lift force is defined, and the interaction between metallic particles is neglected. To estimate the interaction between the particles and the melt flow field, let us consider the following parameters: the particle diameter $d_\mathrm{p} = 0.1\,\mathrm{mm}$ for each metallic element, the characteristic flow velocity $u \approx 1\,\mathrm{m/s}$, the dynamic viscosity of the liquid phase $\mu = 4.5\,\mathrm{mPa{\cdot}s}$ and the characteristic length $L \approx 0.1\,\mathrm{m}$. In this case, the dimensionless Stokes number

$$\mathrm{St} = \frac{\rho_\mathrm{p} d_\mathrm{p}^2 u}{18 \mu L} \tag{3}$$

assumes the values of $\mathrm{St_{Hf}} \approx 0.16$ for hafnium with the highest density, and $\mathrm{St_{Al}} \approx 0.0016$ for aluminium with the lowest density. Intermediate values are calculated for Cr and Ti based on the properties listed in Table 1. It can be stated that the particles are, therefore, fully affected by the liquid flow field. The trajectory of discrete phase particles is predicted by the force balance equation (per unit particle mass):

$$\frac{\mathrm{d}\mathbf{u}}{\mathrm{d}t} = F_\mathrm{D}(\mathbf{u} - \mathbf{u}_\mathrm{p}) + \frac{\mathbf{g}(\rho - \rho_\mathrm{p})}{\rho_\mathrm{p}} + \frac{\nabla p}{\rho_\mathrm{p}} + \mathbf{f}_{\mathrm{p,EM}}, \tag{4}$$

where $\mathbf{u}_\mathrm{p}$ is the particle velocity, $\mathbf{g}$ is the acceleration of gravity and p stands for the pressure. The drag force per unit particle mass $F_\mathrm{D}(\mathbf{u} - \mathbf{u}_\mathrm{p})$ includes the velocity of the *continuum* $\mathbf{u}$ and reads as

$$F_\mathrm{D} = \frac{18 \mu C_\mathrm{D} \mathrm{Re}}{24 \rho_\mathrm{p} d_\mathrm{p}^2}. \tag{5}$$

The relative Reynolds number of the particle Re is expressed as

$$\mathrm{Re} = \frac{\rho d_\mathrm{p} |\mathbf{u}_\mathrm{p} - \mathbf{u}|}{\mu}. \tag{6}$$

Table 1. Material properties used for particle tracing computation in the fluid dynamic model. Properties are referred to 1835°C, the measured temperature of melting of the NbSi-based composite.

Material property	**Al**	**Ti**	**Cr**	**Hf**	**Nb**	**Si**	**NbSi**
Electrical conductivity, [MS/m]	2.5	0.606	0.667	0.592	1.388	6.579	1.388
Mass density, [kg/m^3]	2698.9	4850	6350	13280	8570	2500	7160
Specific heat, [J/kg·K]	897	930	544.25	140	265	700	260

The buoyancy force on a solid particle is defined as the ratio between the gradient of the pressure field in the melt ∇p and the material density of the particle ρ_{p} [16].

Electromagnetic forces acting on metallic particles include Leenov–Kolin forces. For spherical particles, they are defined as

$$\mathbf{f}_{\mathrm{p,EM}} = -\frac{3}{2}\frac{\sigma - \sigma_{\mathrm{p}}}{2\sigma + \sigma_{\mathrm{p}}}\frac{1}{\rho_{\mathrm{p}}}\mathbf{f}_{\mathrm{EM}}, \tag{7}$$

where σ is the electrical conductivity, ρ_{p} is the mass density of the particle material, and $\mathbf{f}_{\mathrm{EM}} = 0.5\Re\mathrm{e}\{\mathbf{J} \times \mathbf{B}^*\}$ is the average component of the Lorentz force generated by the EM field.

2. Melting and alloying of NbSi-based composite.

Multiple samples of niobium and silicon have been already obtained by the authors: the obtained alloy was binary and eutectic, and the measured temperature at the melting point was 2040°C [10]. The power supplied by the generator was relatively low (50 kW) if compared with the one necessary to melt pure niobium (113 kW). The binary alloy was melted in a vacuum atmosphere, and the process was initiated from the pellets of the single elements to ensure the electromagnetic coupling with the operating frequency and maximize the mixing of niobium and silicon inside the compound. The experimental results with the niobium-silicon alloy were in good agreement with the numerical simulation in terms of the power supplied to reach the melting temperature of the alloy and the generated amounts of liquid and solid fractions [10, 11]. The variety of experienced melting conditions evidenced of the most stable fluid dynamic and thermal behaviour for the sample with the volume 130 cm^3. The future investigation steps will be based on those conclusions.

2.1. Alloying of six components: numerical results. This work starts with the inclusion of additional elements – aluminium, titanium, chromium and hafnium – to the obtained NbSi base. The targeted global volume of the melt was 130 cm^3. The metals were modelled as spherical, solid particles, with the same diameter of the powders used for the experimental investigations. A group counting ten particles for each material was selected and injected into the liquid phase at the height of $Y_{\mathrm{in}} = 20$ mm from the bottom of

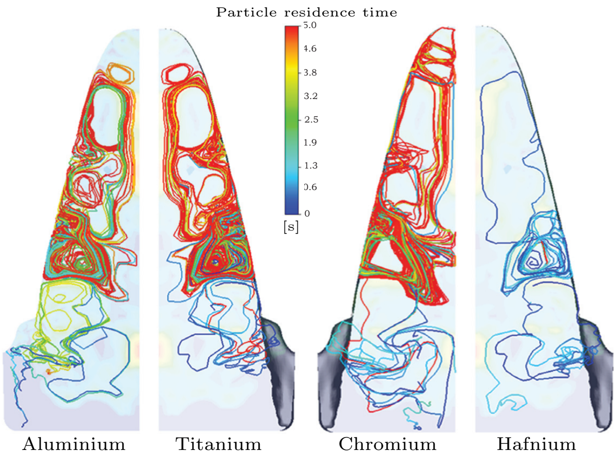

Fig. 4. Computational results obtained by the particle tracing method after the simulation time $t_{\mathrm{sim}} = 12$ s: motion of the additional metals. Particle tracks are plotted in terms of the residence time.

the crucible. The injection position was determined as a result of the manual optimization for the mixing effect of the elements; the achieved strong stirring was supposed to disperse them uniformly in the final cast. The numerical model considers a *continuum* of NbSi, in which the motion of the additional metals is affected by different forces. Because of the significant lack of the literature, some of the properties of the NbSi composite were determined on the base of experimental results, in particular, the melting point of the NbSi base which was initially set as 2040°C and iteratively corrected based on the experimental results. Numerical simulations were performed for the total time of $t_{\mathrm{sim}} = 12\,\mathrm{s}$. The final results are plotted as tracks patterned by the particles and their residence time (Fig. 4): the residence time expresses the time occupied by a particle inside a certain control volume. It is evidenced that aluminium, titanium and chromium tend to distribute in the upper region of the melt volume, transported by the typical recirculation vortices of the flow field. Chromium occupies the peak of the liquid workpiece for a residence time which overcomes 5 s and is driven by larger vortices compared to the other materials. This effect cannot be recognized in the case of aluminium even if its mass density is significantly lower; in fact, a stronger motion towards the top of the melt was expected. The dispersion of aluminium could be explained in detail: a smaller residence time of its traces indicates a faster recirculation of aluminium which partially distributes even in the lower region of the liquid pool. Finally, the distribution of hafnium is very inhomogeneous and actually affected by its high mass density. The stronger recirculation of its particles is visible in the central region of the melt since they are possibly trapped by the flow field.

2.2. Alloying of six components: experimental results. The experimental investigation was carried out starting from the inclusion of powders of the additional elements into the pellets of niobium and silicon: similarly to the numerical model, a layer of additional metals was placed at the height of $Y_{\mathrm{in}} = 20\,\mathrm{mm}$ from the bottom; their global volume was minimum in comparison with the components of the NbSi base. The melting process was carried out once in the vacuum atmosphere. The experimental results evidence of partial melting of the NbSi base with the additional elements; the registered electrical parameters were the power supplied by the generator $P_{\mathrm{gen,exp}} = 36\,\mathrm{kW}$ at the melting point, the inductor current $I_{\mathrm{ind,exp}} = 1418\,\mathrm{A}$ rms and the working frequency $f_{\mathrm{exp}} = 10.9\,\mathrm{kHz}$. The temperature of the workpiece measured at the melting point was 1835°C and the liquid melt maintained a stable meniscus. The registered melting point was corrected in the numerical model and adopted for future numerical investigations.

Yet, the chemical analysis reported the incomplete alloying of the materials: in contrast to the numerical results, hafnium fully melted inside the NbSi base and distributed uniformly in the final alloy. The mixing of titanium was not homogeneous, and this metal outlines the recirculation patterns still visible in the upper region of the solidified cast. Such vortices are analogous to those calculated numerically and show a good agreement between the experiment and the model (Fig. 5). The numerical simplification with half palisade is supposed to be responsible for the asymmetrical behaviour of the flow patterns of titanium. The analysis reported a heavy evaporation of aluminium and chrome: the atomic percentage of Al remaining inside the solid cast was 0.8%, whereas its initial value was 3%; the remaining atomic percentage of Cr was 0.3%, whereas its initial value was 4%. Such intense evaporation is explained by the vacuum environment in which the melting process takes place: this investigation was carried out under a pressure of 10^{-2} mbar, which dramatically decreased the boiling point of the targeted materials. Finally, the qualitative analysis of the sample showed unmelted pellets of niobium in the lower region of the cast. Their presence can be explained by the combination of unfavorable melting conditions like the extreme melting point of niobium and the lower temperature in the proximity of the solid skull.

2.3. Improvement of the alloying process using argon atmosphere. Basing on the previous results, the following experiments were carried out in modified melting conditions: the protective chamber was filled with argon to create an inert environment

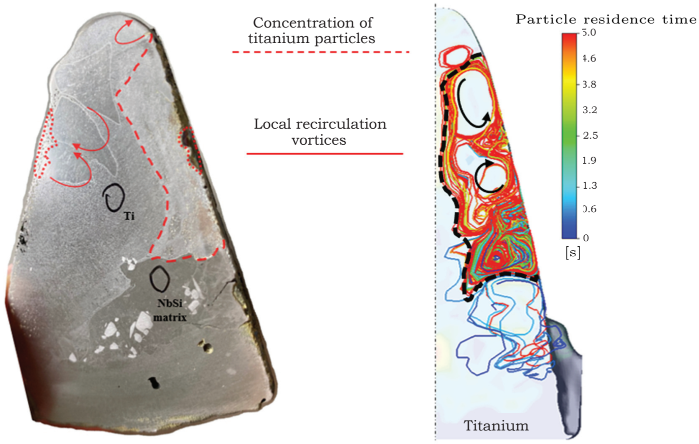

Fig. 5. Comparison between the first experimental result (left) and the numerical result with particle tracing (right). The lower volume of the solidified sample mostly consists of the $Nb_{18}Si$ matrix, hafnium and 20% of titanium; the region can be recognized by its dark grey colour, and some unmelted pellets of niobium are still visible. The upper volume consists mostly of titanium (80%) and a limited amount of the $Nb_{18}Si$ matrix; the presence of titanium is evidenced by light grey colour. The distribution of titanium is marked by its recirculation patterns: the cast and the numerical result evidence of significant similarities.

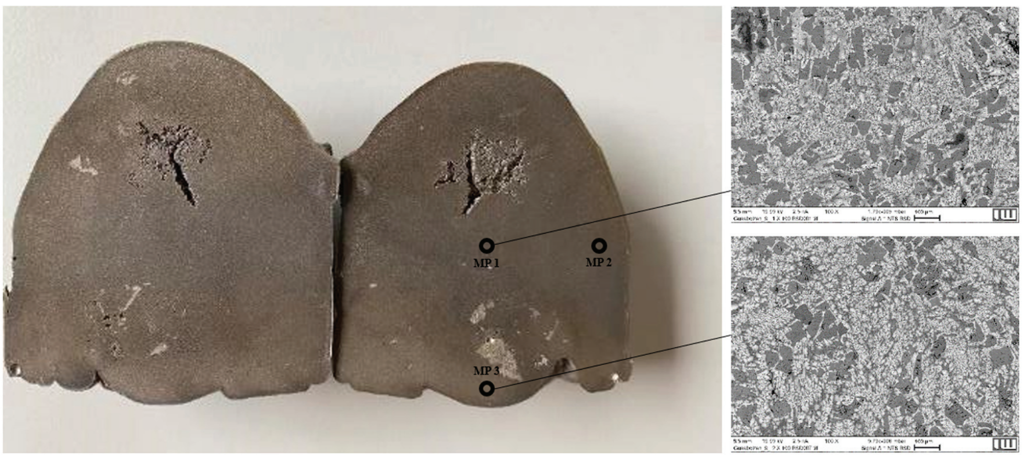

Fig. 6. Experimental result on the second melt: the solidified sample cut in two pieces (left) and its microscopic structure at two points. The black circles show the measuring points of the cast composition (MP no. 1, 2 and 3). Source: Institut für Werkstoffkunde (IW) of the Leibniz University Hanover.

Table 2. Experimental results: composition of the elements in at.%. The sample was cut in the middle, and its composition was measured at three measuring points (MP) as indicated in Fig. 6. The measured composition is compared to the targeted one in the case of perfectly uniform distribution.

Material	MP nr.1	MP nr.2	MP nr.3	Target
Aluminium (Al)	4.01	3.96	4.16	3
Titanium (Ti)	22.14	23.01	21.95	22
Chromium (Cr)	3.99	4.41	3.96	4
Hafnium (Hf)	1.69	1.68	1.57	2
Silicon (Si)	16.62	15.76	17.58	16
Niobium (Nb)	51.56	51.19	50.78	53

at a constant pressure of 300 mbar. The melting process was repeated twice for the same sample after rotating the solid workpiece inside the ICCF. In this way, homogenization of materials was expected to improve if compare to the first experiment.

The chemical analysis reports that all additional metals alloy with the base of Nb and Si with no evaporation of any of them (Fig. 6). All materials distribute homogeneously within the solid cast, as summarized in Table 2. Insignificant discrepancies are reported for niobium and aluminium: the former showed the tendency to better homogenize in the lower region of the cast, the latter one near the sides of the composite was probably transported by a stronger flow near the free surface.

3. Discussion and conclusions.

The melting process in the cold crucible induction furnace for a composite based on niobium and silicon was numerically and experimentally investigated. Alongside, the alloying process between the NbSi base and additional elements – aluminium, titanium, chromium and hafnium – was analysed with the help of numerical models and examination of the compound. The obtained computational results showed a good agreement with the fluid dynamic and thermal behaviour of the melting, confirming the prediction of the experimental conditions. Despite the large difference between their material properties, all elements were successfully molten with a low fraction of the maximum available electric power. The volume of 130 cm^3 for the sample was confirmed to be the best compromise between the stable melting conditions and the amount of the product.

The first alloying experiment revealed a strong inhomogeneous mixing of the NbSi with the additional elements that was affected by the evaporation of aluminium and chromium. The unsuccessful results can be explained by the use of vacuum atmosphere and the fact that an attempt to melt the sample was made only once. The distribution of the metallic components was predicted with good reliability by the discrete phase model: the particle tracing method was applied as a supporting tool to analyse their combination with the compound and to optimize the alloying process.

The second melting was performed twice for the same sample and in the argon atmosphere under a pressure of 300 mbar: the analysis of the solid compound evidenced of the generation of a homogeneous alloy, where all additional elements were mixed inside the NbSi base without the evaporation effect. Improvements of the mechanical properties of the cast are expected due to the significant modification of the microstructure of the composite.

The successful achievement of the six-elements' NbSi-based composite in ICCF represents a further advance in the production of chemically reactive alloys with a high melting point. It also foreshadows a suitable approach for the fabrication of components operating in high-temperature environments. In parallel, advances in numerical computation evidence that the model based on discrete phases can be a reliable instrument to predict and optimize the alloying in induction melting processes.

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