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# EFFECT OF HOLDING TIME ON THE PRODUCTION OF Nb-$NbAl_3$ INTERMETALLIC COMPOSITES VIA ELECTRIC-CURRENT-ACTIVATED SINTERING

## VPLIV ČASA ZADRŽANJA NA IZDELAVO Nb-$NbAl_3$ INTERMETALNIH KOMPOZITOV Z ELEKTRIČNIM TOKOM AKTIVIRANIH S SINTRANJEM

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A recently developed powder metallurgy processing technique – Electric Current Activated (Assisted) Sintering (ECAS) was employed to produce intermetallic Nb-$NbAl_3$ composites. In this study, to produce Nb-$NbAl_3$ in-situ intermetallic composites, Nb (99.8 % purity, less than 44 μm) and Al (99.5 % purity, less than 44 μm) elemental powders were mixed in the stoichiometric ratio corresponding to the Nb-Al phase diagram. The effect of different processing times, for (10, 30, 60) s, under maximum of 2000 A and 1.5-2.0 V, was investigated. Scanning electron microscopy and X-ray diffraction analysis were used to characterize the produced samples. X-ray diffraction studies revealed that the dominant phases are $NbAl_3$ and Nb. Scanning electron microscopy examinations showed a dense microstructure with a very low amount of porosity and also a trace amount of residual aluminium. The microhardness of the test materials sintered for 60 s via electric-current-activated sintering was about 405 HV±46 HV0,05.

Keywords: in situ composites, $NbAl_3$ aluminides, electric current activated sintering (ECAS)

Pred kratkim razvita tehnika v metalurgiji prahov: sintranje, aktivirano z električnim tokom (ECAS), je bilo uporabljeno za in situ izdelavo intermetalnega kompozita Nb-$NbAl_3$. V študiji sta bila za in situ proizvodnjo Nb-$NbAl_3$ intermetalnega kompozita zmešana elementna prahova Nb (99.8 % čistost, delci manjši od 44 μm) in Al (99.5 % čistost, delci manjši od 44 μm), zmešana v stehiometričnem razmerju, skladno s faznim diagramom Nb-Al. Preiskovan je bil vpliv različnih časov izdelave: (10, 30, 60) s pri toku maksimalno 2000 A in napetosti 1,5-2,0 V. Izdelani vzorci so bili karakterizirani z vrstično elektronsko mikroskopijo in rentgensko difrakcijo. Rentgenska difrakcija je odkrila, da sta prevladujoči fazi $NbAl_3$ in Nb. Vrstična elektronska mikroskopija je pokazala gosto mikrostrukturo z majhnim deležem poroznosti in sledovi preostalega aluminija. Mikrotrdota preizkusnega materiala, sintranega 60 s, s sintranjem aktiviranim z električnim tokom, je bila okrog 405 HV±46 HV0,05.

Ključne besede: in situ kompoziti, $NbAl_3$ aluminidi, sintranje aktivirano z električnim tokom (ECAS)

## 1 INTRODUCTION

Intermetallic compounds have been the focus of significant research and development efforts during recent years. Among intermetallic compounds, niobium aluminides are very important and attractive.[1–3] Three intermetallic compounds are present in the Nb–Al binary system including $Nb_3Al$ (A15 structure), $Nb_2Al$ (D8b structure) and $NbAl_3$ (DO22 structure, $TiAl_3$ type).[4–7] Among the various compounds in the Nb-Al system (**Figure 1**), $NbAl_3$ with its high melting point (1680 °C), low density (4.54 g/cm$^3$), is attractive as a potential material for high-temperature applications.[6,8] The applications of $NbAl_3$ include its use in turbine blades in aircraft engines or in stationary gas turbines.[1,9] However, despite its attractive features, its usage is limited by inadequate ductility at room temperature. So, for optimization of room-temperature toughness, microstructural modifications are required.[1,6,10] In-situ toughening is an alternative technique to enable a combination of brittle intermetallic phase with a ductile metallic phase in one step production.[11,12] Some conventional methods such as melting, casting and mechanical alloying techniques or self-propagating high-temperature synthesis (SHS)[7–9] can be used for manufacturing intermetallics. A recently

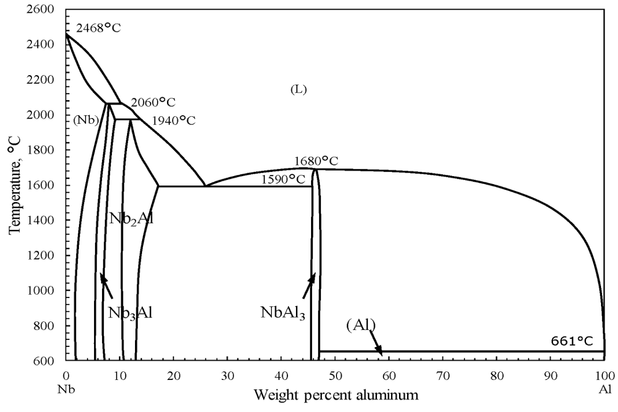



**Figure 1:** Nb-Al phase diagram[7]
**Slika 1:** Fazni diagram Nb-Al[7]

developed electric current activated/assisted sintering (ECAS) technique has been used in this study. This system enables the cold formed compact obtained from uniaxial compression to be inserted into a container, which is heated by passing an electrical current. In the present paper we prefer to apply the direct current resistive sintering technique to the Nb+3Al powder mixture in order to investigate a new route to determine the effect of holding time on the production of Nb-$NbAl_3$ in-situ composites.

## 2 EXPERIMENTAL PART

### *2.1 Materials and methods*

Al and Nb elemental powders with 35–44 μm grain size and purity of 99.5 % and 99.8 %, respectively, were mixed to give the nominal composition of Nb40Al60 (*w*/%) for the formation of a $NbAl_3$ intermetallic based metallic Nb reinforced in-situ composite. The powder mixture was ball milled for 15 min then cold-pressed before sintering to form a cylindrical compact in a metallic die under a uniaxial pressure of 200 MPa. The dimensions of the compact were 15–16 mm diameter and 3–4 mm thickness. The production of Nb aluminide-based intermetallic compound was performed via the electric current activated sintering technique in an open atmosphere at 2000 A for (10, 30, 60) s. They are denoted as C1 and C2 and C3 in the following section, respectively. Th eprocess parameters are listed in **Table 1**.

**Table 1:** Process parameters for the samples
**Tabela 1:** Procesni parametri vzorcev

| Sample code | *w*/% | Current (A) | Holding time (s) |
|---|---|---|---|
| C1 | 40Nb-60Al | 2000 | 10 |
| C2 | 40Nb-60Al | 2000 | 30 |
| C3 | 40Nb-60Al | 2000 | 60 |

### *2.2 Characterization*

The morphologies of the samples were examined by scanning electron microscopy (SEM-EDS) in terms of the resulting phases. X-ray diffraction (XRD) analyses were carried out using Cu–$K\alpha$ radiation with a wavelength of 0.15418 nm over a $2\theta$ range of 10–80°. The microhardness of the test materials was measured using a Vickers indentation technique with a load of 0.98 N using Leica WMHT-Mod model Vickers hardness instrument.

## 3 RESULTS AND DISCUSSION

### *3.1 SEM-EDS Analysis*

**Figure 2** shows SEM micrographs of the elemental Nb and Al powder particles. As seen in **Figure 2** the Al particles are rounded and Nb particles are angular and sharp cornered in shape.

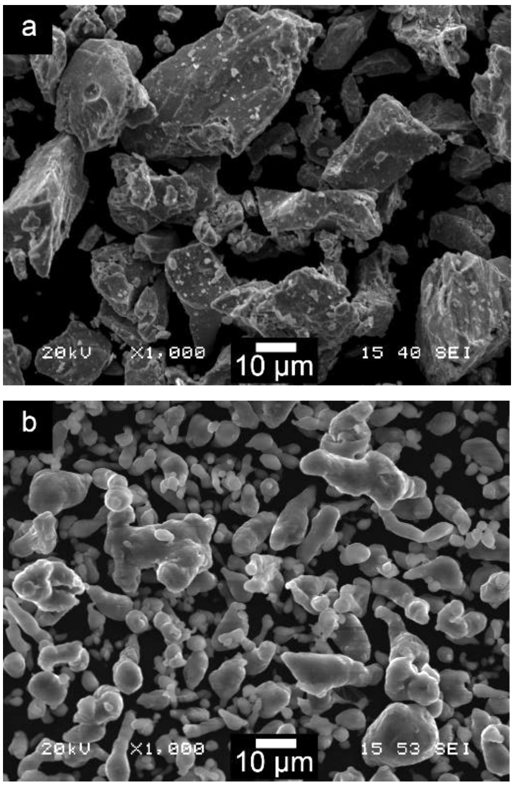



**Figure 2:** SEM micrographs of: a) Nb, b) Al powder
**Slika 2:** SEM-posnetka: a) Nb in b) Al prahu

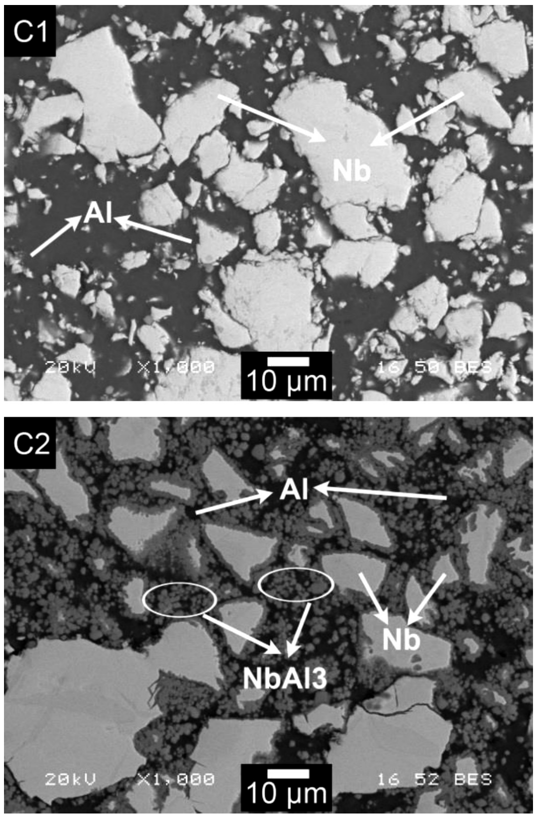



**Figure 3:** SEM micrographs of: a) C1 and b) C2 samples
**Slika 3:** SEM-posnetka vzorcev a) C1 in b) C2

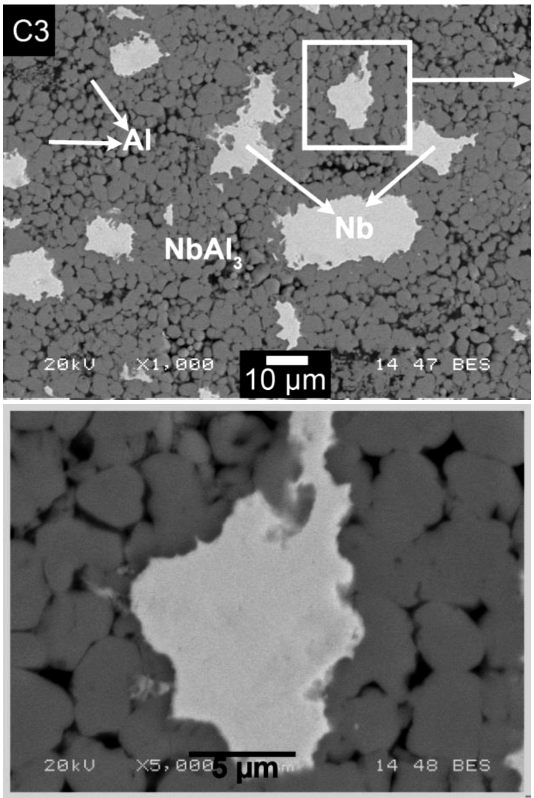



**Figure 4:** SEM micrographs of C3 Sample: a) 1000 ×, b) 5000 ×
**Slika 4:** SEM-posnetka vzorca C3: a) 1000 ×, b) 5000 ×

SEM-EDS analyses of C1, C2 and C3 intermetallic compounds are shown in **Figure 3**. The microstructure in **Figure 3a** shows that the low holding time results in separately formed Nb and Al areas. Increasing the processing time from 10 s to 30 s in C2, (**Figure 3b**), it starts to form a new phase like $NbAl_3$, but these microstructures are still far from the desired stoichiometric composition of the main $NbAl_3$ phase.

When it comes to the C3 sample, as seen in **Figure 4**, increasing the process holding time to 60 s, the main phase in the microstructure is Nb and $NbAl_3$. Besides this, there is also a little amount of residual Al phase and a little oxidation problem because of the open atmosphere sintering in ECAS. But that was not detected in the XRD analyses for being a small amount. In addition to that, a nearly fully dense microstructure was obtained after just a minute of holding process time, thanks to the electric current resistive sintering system.

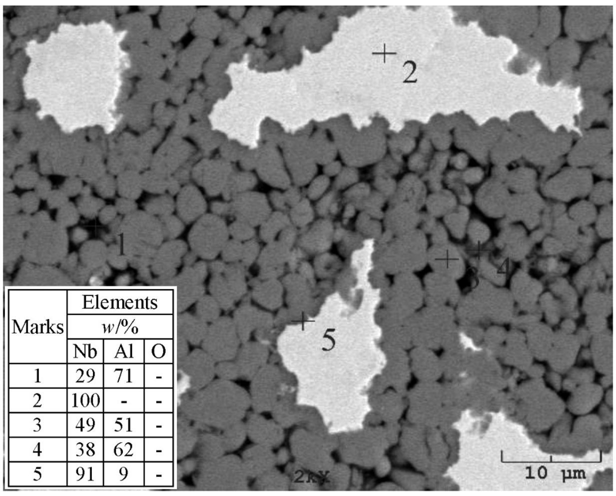
As can be seen in the SEM-EDS analyses in **Figure 5**, the reaction was also not completed in the C3 compound. It is assumed that the applied voltage or current is insufficient for the complete transformation of the $NbAl_3$ phases in the sintering.

### *3.2 XRD Analysis*

The XRD analysis, **Figure 6**, shows that the main phase of the composite is $NbAl_3$. The Nb phase is also seen in the XRD analyses, as desired. These results support the observations from the SEM-EDS analysis (**Figure 5**). This can be inferred from this result: Nb-$NbAl_3$ in-situ composites can be obtained for one minute and one step electric current activated system; however, for eliminating the residual aluminium in the compact. It is obvious that, it can be optimized with some other parameters such as voltage or current.

### *3.3 Hardness*

The hardness values $HV_{0.05}$ of the C1, C2 and C3 samples were measured as 165±20, 250±27, 405±46 respectively. The hardness of the intermetallic composites increases from 165±20 HV to 405±46 HV by increasing the holding time in the process due to the formation of a higher proportion of intermetallic phases. The hardness results for the $NbAl_3$ composite is in agreement with the literature.[13]




| Marks | Elements w/% | | |
|---|---|---|---|
| | Nb | Al | O |
| 1 | 29 | 71 | - |
| 2 | 100 | - | - |
| 3 | 49 | 51 | - |
| 4 | 38 | 62 | - |
| 5 | 91 | 9 | - |

**Figure 5:** SEM-EDS analyses of C3 samples
**Slika 5:** SEM-EDS-analize na vzorcu C3

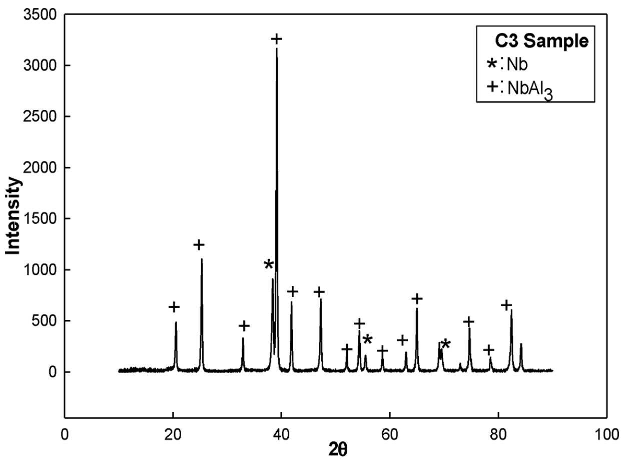



**Figure 6:** XRD Analyses of C3 sample (Nb-$NbAl_3$)
**Slika 6:** Rentgenogram vzorca C3 (Nb-$NbAl_3$)

## 4 CONCLUSIONS

Some of the results obtained from this study can be summarized as follows:

- Niobium aluminide-based composites were fabricated by electric current activated/assisted sintering at 2000 A in only 60 s.
- Under such conditions, the reaction is nearly completed within a very short period of time and the end-product is consolidated to a nearly fully dense microstructure.
- SEM-EDS and XRD analyses showed that 60 s are sufficient for obtaining Nb-$NbAl_3$ phases. However, for eliminating the residual aluminium in the compact it can be optimized some other parameters such as voltage or current.
- Hardness of the intermetallic composites increases from 165±20 HV to 405±46 HV by increasing the holding time in the process by ensuring the formation of a higher proportion of intermetallic phases

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