

## INVESTIGATION ON THE AGING BEHAVIOUR OF THE FUNCTIONALLY GRADIENT MATERIAL CONSISTING OF BORON CARBIDE AND AN ALUMINUM ALLOY

### RAZISKAVA PONAŠANJA PRI STARANJU FUNKCIONALNIH GRADIENTNIH MATERIALOV IZ BOROVEGA KARBIDA IN ALUMINIJEVE ZLITINE

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In the current study the effect of different temperatures on the aging behaviour of a functionally gradient material was investigated to observe the variation of hardness with respect to the aging time. To do this, the functionally-gradient-material (FGM) specimens, containing boron carbide and aluminum alloy, were produced with hot pressing. Three different layers were used in the FGM samples. Then the macro and micro examinations were carried out to observe the interface between the layers, the porosity, the grain size and probable cracks in these samples. After that the specimens were solutionized at 470 °C for 1 h. Next, the artificial aging at 100 °C, 120 °C and 150 °C was applied to the FGM specimens for 96 h. During the aging treatment, the Brinell hardness measurements were made on the samples at certain intervals. Moreover, three-point bending tests were also carried out to clarify the influence of the aging treatment on the strength of the FGM. Experimental results indicated that the highest hardness values were obtained at 120 °C after the aging period of 48–65 h. The aged specimens exhibited higher bending strength than the solutionized specimens.

Keywords: metallic materials, inorganic materials, aging, three-point bending test

V tej študiji je prikazan vpliv različnih temperatur na staranje funkcionalnega gradientnega materiala in spreminjanje trdote v odvisnosti od časa staranja. Vzorci funkcionalno gradientnih materialov (FGM), ki vsebujejo borov karbid in aluminijevo zlitino, so bili izdelani s stiskanjem v vročem. Trije različni sloji so bili uporabljeni za FGM-vzorke. Na vzorcih so bile izvršene makro- in mikropreiskave stikov med sloji, določene so bile poroznost, velikost zrn in prisotnost morebitnih razpok. Potem so bili vzorci 1 h raztopno žarjeni na 470 °C, nato pa umetno starani 96 h na temperaturah 100 °C, 120 °C in 150 °C. Med postopkom staranja so bile v določenih intervalih izvršene meritve trdote po Brinellu. Dodatno je bil izvršen še tritočkovni upogibni preizkus, da bi opredelili vpliv staranja na trdnost FGM. Rezultati preizkusov kažejo, da je bila dosežena najvišja trdnost pri staranju med 48 h in 65 h na temperaturi 120 °C. Starani vzorci so pokazali večjo upogibno trdnost kot raztopno žarjeni vzorci.

Ključne besede: kovinski materiali, anorganski materiali, staranje, tritočkovni upogibni preizkus

## 1 INTRODUCTION

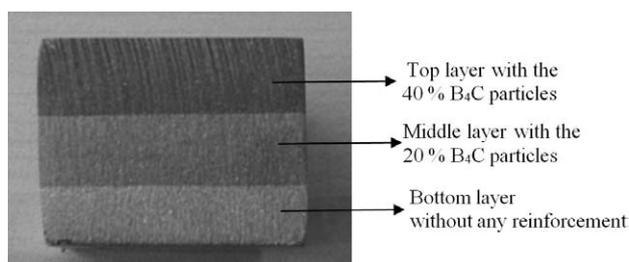
A new type of material called functionally gradient material (FGM) has been considered to be used for special purposes. In this type of material, at least two different types of layers are chemically used in a gradient form. In this way the properties change throughout the cross-section of the material. By definition, an FGM consists of one material being on one side and another material on the other side<sup>1-21</sup>. FGMs have been used in structural, electrical, chemical, optical, nuclear and biomedical applications<sup>1</sup>. The main methods for producing these materials are powder metallurgy, thermal spraying, coating, laser coating and other thermo-mechanical processes<sup>1,2</sup>. Among these methods, powder metallurgy techniques are very suitable for the FGM production<sup>1,2</sup>. The main advantages of chemically forming a gradient in a material are the creation of a metallurgical bond between the layers and different mechanical, nuclear, electrical and/or thermal properties of individual layers<sup>1-20</sup>. Producing a hard outer layer and

a soft inner layer in the same material has been of key importance in the armour applications<sup>1-3</sup>. Therefore, the FGMs containing a hard front layer and a tough inner layer can be suitable for these applications. However, there is a large gap in the literature related to the production and mechanical properties of FGMs that need to be investigated extensively with respect to the objectives of materials science and manufacturing methods. In this paper an FGM with three layers, chemically changing from the top to the bottom, was produced using the powder metallurgy technique. In this material the bottom layer was chosen to be the aluminum alloy (AA) 7075, whereas the boron-carbide particles reinforcing the AA 7075 alloy were used, in different proportions, for the middle and the top layers. The influence of the artificial-aging treatments on the hardness of the layers was examined at different aging temperatures of 100 °C, 120 °C and 150 °C. In addition, the bending strength of the FGMs was determined for the solutionized and the aged samples.

## 2 EXPERIMENTAL PROCEDURE

The FGM samples with three layers were produced with the powder metallurgy technique. The powders used as starting materials were supplied from the market. The mean powder sizes of aluminum, boron carbide, chromium, copper, iron, silicon, zinc and magnesium were measured to be 10.22  $\mu\text{m}$ , 4.04  $\mu\text{m}$ , 33.42  $\mu\text{m}$ , 19.17  $\mu\text{m}$ , 6.66  $\mu\text{m}$ , 3.53  $\mu\text{m}$ , 6.7  $\mu\text{m}$  and 45.08  $\mu\text{m}$ , respectively. Furthermore, the powders had very high purities alternating between 99.5 % and 100 %. In the investigated aluminum alloy, the nominal chemical composition was taken as mass fractions 5.5 % Zn, 2.5 % Mg, 1.6 % Cu, 0.5 % Fe, 0.4 % Si, 0.23 % Cr. The powder size is very important when considering the sintering operation. A small powder size results in a higher surface area, which effectively enhances the sintering operation<sup>22</sup>. The FGM samples were based on the AA 7075 that can be hardened to a very high level by an artificial aging treatment<sup>23-25</sup>.

In the investigated FGM samples, the bottom layer was considered to be a monolithic AA 7075 layer, while 20 % and 40 % B<sub>4</sub>C (in volume) of the particle reinforcements in the AA 7075 matrix were used in the middle and top layers, respectively. Initially, the layers in the suitable chemical proportions were pre-shaped separately by cold pressing. Then they were transferred into the mould of a uni-axial hot press to be sintered at 590 °C for 20 min. After the sintering operation, the artificial aging treatments were performed to get the hardness-aging time curves for the produced FGM. Before the aging process, the samples were solutionized at 470 °C for 1 h and water quenched at 20 °C. The aging temperatures were 100 °C, 120 °C and 150 °C. Furthermore, the macro and the micro examinations on the samples were done to observe probable production defects such as cracks, pores and separations between the layers, as well as distribution of ceramic particles in the matrix. For the metallographic analysis, the samples were prepared with the standard methods and etched using the etchant, a modified Keller's reagent. The hardness tests and the three-point bending tests were made on the solutionized and peak-aged samples according to the standards of ASTM E 10-84<sup>26</sup> and ASTM B 528-05<sup>27</sup>, respectively. For the hardness testing, the



**Figure 1:** Typical cross sectional view of a produced FGM sample with the total thickness of 15 mm

**Slika 1:** Značilen prerez FGM-vzorca s skupno debelino 15 mm

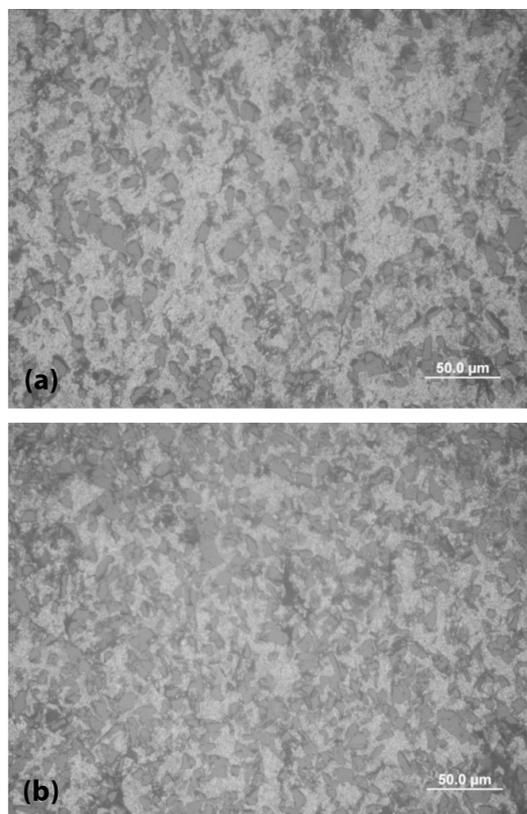
samples were taken out of the furnace and water quenched for the hardness measurement at certain intervals during the aging treatment (every half an hour during the first 6 hours and every hour during the period between the 7th and the 96th hour). Each time the mean values of five measurements were recorded.

## 3 RESULTS AND DISCUSSION

### 3.1 Macro- and microstructural observations

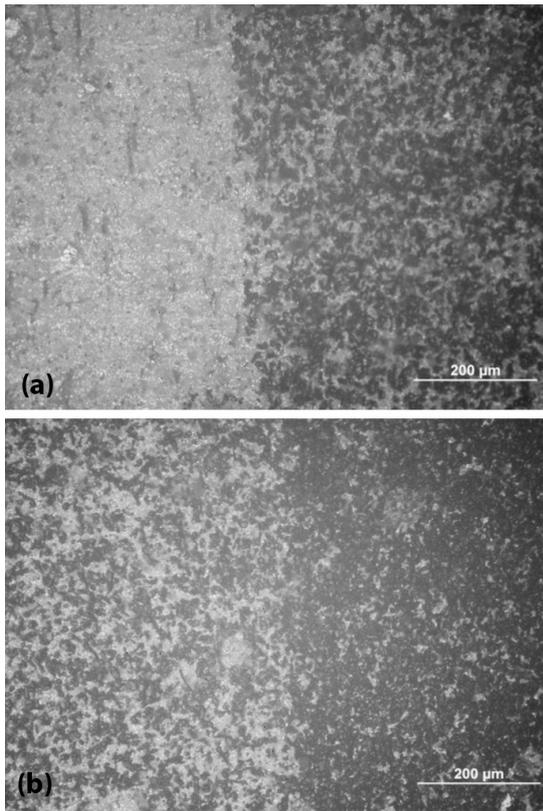
**Figure 1** shows a typical macro view of the cross section for a produced sample with the total thickness of 15 mm. The three layers can be seen clearly in this figure. There are no macro-level cracks or separations observed between the layers of the produced sample. The adhesion of the layers with various compositions appears to be smooth. **Figures 2** and **3** illustrate the microstructure of the produced FGM taken by an optical microscope. A uniform distribution of ceramic particles can be observed on these figures. In addition, there is a good transition-and-interface formation between the layers.

Although the formation of interfaces and the distribution of ceramic particles throughout the samples are found to be uniform, some porosity is detected. The porosity level, detected by an image-analysis program, is estimated to be 5 %, 6 % and 8 % for the bottom, middle and top layers, respectively. Moreover, the average grain



**Figure 2:** Micro view of: a) the middle and b) the top layers

**Slika 2:** Mikroposnetek: a) sredine in b) vrhnjih plasti



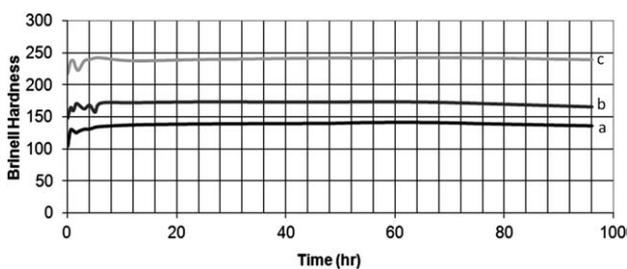
**Figure 3:** Interfaces between: a) the bottom and the middle, b) the middle and the top layers

**Slika 3:** Stik med: a) spodnjo in srednjo, b) srednjo in zgornjo plastjo

size was measured to be 24 µm according to the ASTM E 112<sup>28</sup>.

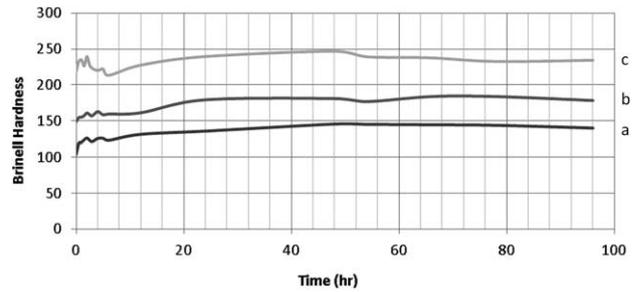
### 3.2 Aging effects on the hardness

**Figure 4** gives the hardness/aging time curves for the sample aged at 100 °C. Initially, the hardness values for the bottom, middle and top layers are HB (104, 148 and 218), respectively. The hardness of all the layers increases after 30 min. Later there are some fluctuations in the curves in the aging period of the first 6 h. Finally, the hardness is stabilized for all three layers sometime between the 6th and the 96th hour. The highest hardness



**Figure 4:** Hardness versus the aging time at 100 °C for: a) bottom, b) middle and c) top layers

**Slika 4:** Trdota v odvisnosti od časa staranja pri 100 °C za: a) spodnji, b) srednji in c) zgornji sloj



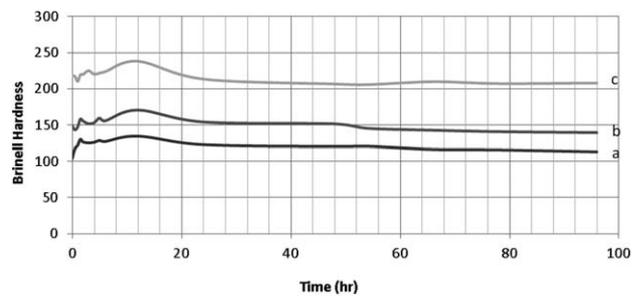
**Figure 5:** Hardness versus the aging time at 120 °C for: a) bottom, b) middle and c) top layers

**Slika 5:** Trdota v odvisnosti od časa staranja pri 120 °C za: a) spodnji, b) srednji in c) zgornji sloj

values recorded for the bottom, middle and top layers are HB (142, 172 and 242), respectively.

The aging curves for the sample aged at 120 °C are given in **Figure 5**. As expected, the highest hardness value belongs to the top layer. The general tendency in the shape of the curve is very similar for all three different layers. After the solutionizing treatment, the hardness is measured to be HB (104, 149 and 218) for the bottom, middle and top layers, respectively. During the aging period of the first 4–5 h, there are some fluctuations in the hardness profile. However, later the hardness starts to increase reaching the peak levels between the 48th and the 66th hour for all the layers. During the period between the 66th and the 96th hour, a gradual reduction in the hardness is observed due to over aging. The highest hardness values measured for the bottom, middle and top layers are HB (145, 181 and 247), respectively.

**Figure 6** depicts the change in the hardness of the sample with respect to time at the aging temperature of 150 °C. As in the aging treatments at 100 °C and 120 °C, there is no change in the hardness profile of the alloy including the B<sub>4</sub>C ceramic particles. After the aging period of 12 hours, the hardness is recorded to be HB (135, 171 and 239) for the bottom, middle and top layers, respectively. The hardness begins to decrease after the 12 h aging period. At the end of the 66th hour, the hardness becomes HB (116, 142 and 210) for the same layers, respectively. When aging the sample at 150

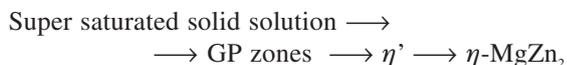


**Figure 6:** Hardness versus the aging time at 150 °C for: a) bottom, b) middle and c) top layers

**Slika 6:** Trdota v odvisnosti od časa staranja pri 150 °C za: a) spodnji, b) srednji in c) zgornji sloj

°C, the peak hardness values are obtained in shorter times, but they are lower than those obtained at the temperature of 120 °C.

The AA 7075 is a heat-treatable alloy<sup>23</sup> and its precipitation sequence can be given as follows<sup>24</sup>:

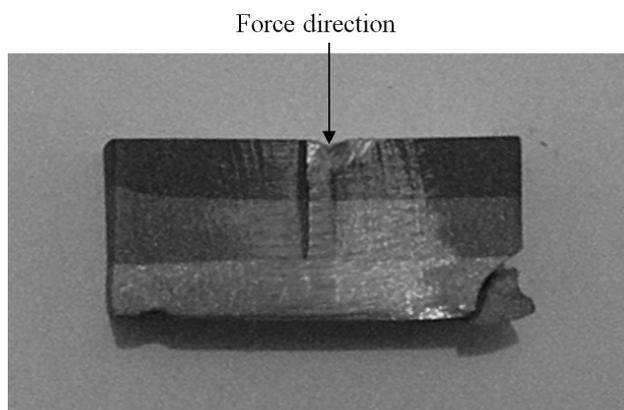


It is considered that the highest hardness is reached when the metastable phase of  $\eta'$  is formed during the aging<sup>23,25</sup>. The GP zones and  $\eta'$  form at the beginning of the aging treatment. The size and density of the precipitates very much affect the mechanical properties<sup>23,25</sup>. In the current work the higher hardness levels are obtained at the aging temperature of 120 °C. Therefore, it is thought that an extensive formation of the  $\eta'$  phase takes place at this temperature. Although the precipitation kinetics seem to be higher at 150 °C, the hardness values recorded at this temperature were lower than those obtained at 120 °C.

In a previous study<sup>29</sup> on the aging of AA 7075, the peak hardness value ( $\approx$ HV 190) was reached at 121 °C and after 48 h. The general trend of the hardness profile and the hardness values found in this study are similar to those found in ref.<sup>29</sup>. Furthermore, in another study<sup>30</sup> the highest hardness value was recorded to be HV 170 at 120°C after 48 h for the AA 7075 produced with powder metallurgy. This result is also consistent with the current data given in this work.

### 3.3 Bending strength

During the bending test the force was applied through the layer with the 40 % B<sub>4</sub>C particle reinforcement. The bending strength of the solutionized sample was found to be 456 MPa, whereas the bending strength of the aged sample was measured to be 527 MPa. A remarkable effect of the aging on the bending strength of the FGM was observed. During the testing a major crack was formed through the main axis that was exposed to the



**Figure 7:** Macro view of the non-aged sample with the total thickness of 15 mm after the three-point bending test

**Slika 7:** Makrovidez nestaranega vzorca s skupno debelino 15 mm po tritočkovnem upogibnem preizkusu

bending force and some particle fractures occurred on the bottom layer of the non-aged sample that has lower hardness (**Figure 7**). On the other hand, no big crack formation was observed on the aged sample, as only a fracture of some small particles were detected on its bottom layer. The rigidity of this sample increased with an increase in its hardness.

## 4 CONCLUSIONS

The peak hardness values for all the layers of the investigated FGMs were found at the aging temperature of 120 °C. The artificial aging of the FGM at 120 °C allowed about 40 %, 21 % and 13 % increase in the hardness of the bottom, middle and top layers, respectively. The addition of the B<sub>4</sub>C ceramic particles to the AA 7075 matrix had no significant effect on the aging behaviour of the alloy. The aged specimen exhibited a 15 % higher bending resistance than the non-aged one. In order to enhance the hardness and strength of the investigated FGMs, the porosity level should be reduced or eliminated using higher pressure and smaller powder sizes.

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