

FGMs generated method SHSM

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Abstract

In this study, a new SHSF (Self-Propagating High-Temperature Synthesis in Mould) process of synthesis of the functionally gradient materials (FGMs) was described. The said method enabled generating a thin, sub-surface layer of the MMC type in two alloys, i.e. in cast iron/NbC and Ni₃Al/NbC systems. The layer was composed of fine-dispersed niobium carbides present in high volume content. The cast iron/NbC and Ni₃Al/NbC materials fabricated by the SHSF process were next subjected to metallographic and structural examinations, combined with testing of mechanical properties.

Keywords: Functionally gradient materials; SHS, SHSB, Intermetallic phase, Iron, Metal carbide NbC

1. Introduction

Owing to a unique complex of properties and unconventional methods of fabrication, functionally gradient materials (FGMs) are nowadays in the centre of interest of materials science. The possibilities to apply these materials in modern technique seem to be practically unlimited, including – among others – the engineering industry, automotive parts, aircraft, optical industry, power engineering, electronics, and numerous fields of physics and medicine. The main parameter used in description of these materials is anisotropy of their chemical composition and structure observed in some areas of the product, mainly in its subsurface layers [1]. The synthesis of these materials results in the formation of functionally gradient structure, characterised by physico-chemical and mechanical properties changing gradually with an increasing distance from the surface.

The main advantage of these materials is controlled transition of properties, different in the „core” of the product and in its surface layer. An effect of this type can be either continuous or changing in a stepwise mode and characterised by a narrow gradient zone boundary.

At present, numerous techniques are available and used in the fabrication of functionally gradient structures [1-4]. The best known include the techniques of plasma spraying [1,5], laser cladding, infiltration of performs [1], sedimentation deposition [1], consolidation of gradient materials (pressing, sintering, SHS) [1,6] chemical vapour and physical vapour deposition (CVD and PVD,

respectively) [4]. Casting techniques are also used, e.g. Tape Casting, Centrifugal Casting and Sedimentation Casting [1]. Owing to their specific characteristics, all these methods enable fabrication of a final product of the predetermined properties. The range and frequency of application of a given method in the fabrication of FGMs depend to a great extent on the economic aspects of the process. Therefore, searching for new techniques in the manufacture of gradient materials should rather tend towards the low-cost and high-output processes.

Within the scope of this research, for the first time, a new pioneer technique of the fabrication of functionally gradient materials was presented. This is the SHSM technique (Self-Propagating High-Temperature Synthesis in Mould), which consists in the “in situ” process of composite fabrication by the SHSB [7-8] route combined with common casting technology. A characteristic feature of this technique is that the process of the formation of a gradient structure takes place in parallel with shaping of the fabricated material into a final product.

2. Methods of investigation

The manufacturing process of the new gradient materials required the application of two different techniques and was proceeding at two stages. The first stage included preparation of moulds, the walls of which were coated with a reactive layer using two methods for the coating deposition. The first method consisted in consolidation of the layer on the mould bottom and side walls.

The second method used mould “washing” for the deposition of this layer. The reactive layer was composed of substrates necessary to initiate the SHS reaction [9]. The mould washing technique required previous preparation of metallic powder suspension (in this research it was powdered niobium of 44 micrometers particle dimensions) and non-metallic powder suspension (powdered coal of the same gradation), mixed in appropriate ratios and suspended in a liquid. As a base material in which the gradient structure should be produced, two base alloys of different properties were selected. It was the cast iron and Ni-Al alloy solidifying as an intermetallic phase of Ni_3Al .

Melting was carried out in a vacuum furnace made by Balzers, using vacuum of 10^{-3} MPa. Molten alloys were next cast into the previously prepared ceramic moulds. The iron was cast into a mould coated with the condensed stoichiometric mixture of Nb and C powder; the Ni_3Al alloy was cast into a mould previously washed with a suspension of powders containing substrates of the products, which enabled the spontaneous synthesis (SHS) of niobium carbides to be initiated and developed. After knocking out the ready iron/NbC and Ni_3Al/NbC castings from moulds, samples were taken for the metallographic and structural examinations and for mechanical testing. The results of the investigations are plotted in Figures 1-10 and compared in Tables 1-2.

3. The results

Figure 1 shows the cross-section of a specimen taken from the cast iron/NbC material. In upper part of the casting one can see a light-colour layer indicating the presence of an area characterised by the composition different than the remaining part of casting.

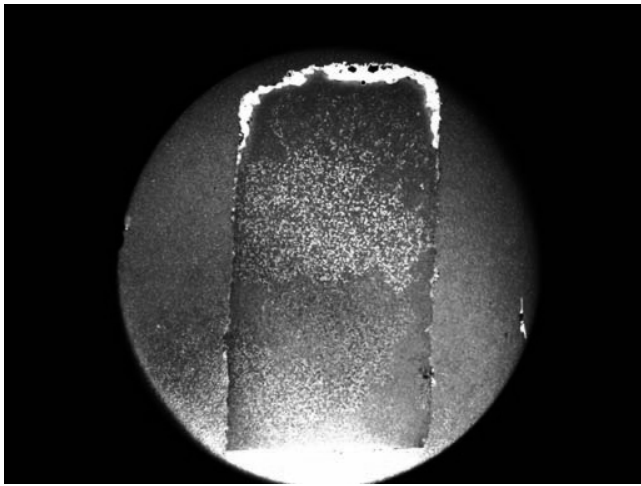
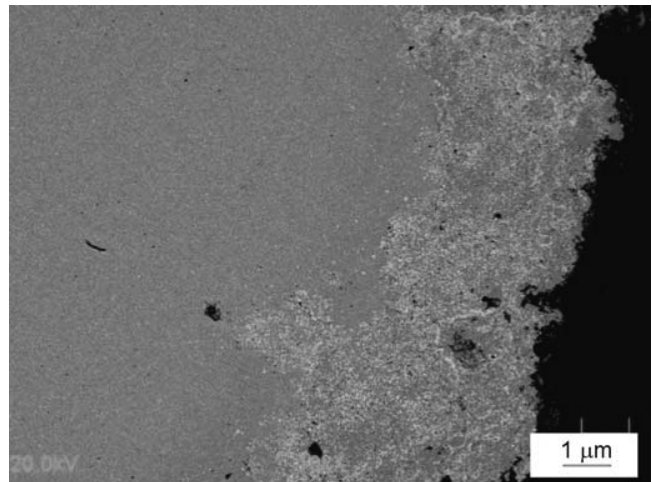


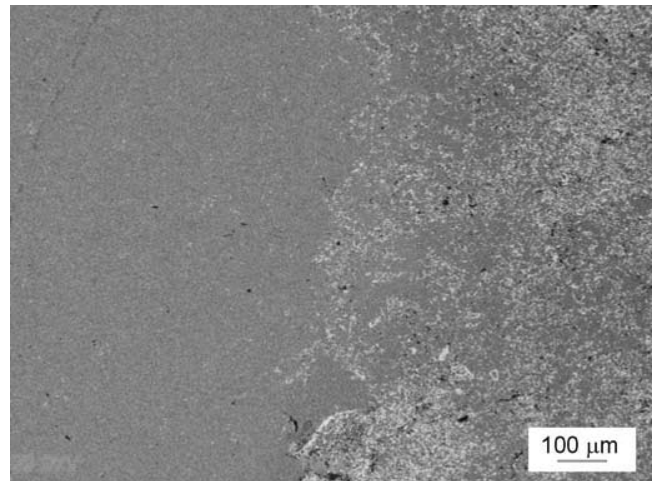
Fig. 1. The macrostructure of cast iron/NbC gradient material. Longitudinal section of the cast cylinder etched with *aqua regia*, magn. 3,15x

To reveal the morphology and chemical composition of the produced layer, scanning examinations and X-ray microanalysis were made. Figure 2 shows scanning electron images of the examined material with a well visible two-phase and gradient structure. Figure 2a shows the casting skin with well visible roughness and small porosity, revealed after the casting was knocked out from mould; the porosity was caused by a coarse-grained mould material. The next drawings, i.e. Figure 2b, show

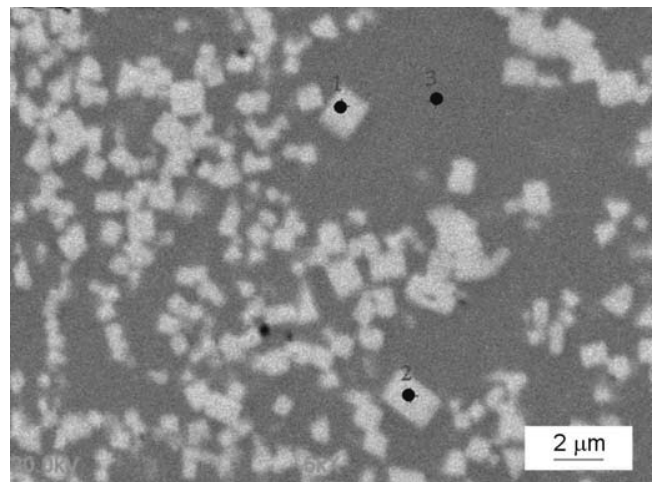
gradual and continuous change of structure, due to the decreasing content of one of the phases.



(a)



(b)



(c)

Fig. 2. The structure of cast iron/NbC gradient material (a-b) with marked points of X-ray microanalysis (c)

The X-ray microanalysis, the results of which are compared in Table 1, was conducted at points marked in Figure 2c. It has proved that the, generated in cast iron matrix, phase deciding about the gradient properties of the material contains Nb and C (points 1 and 2, respectively, in Figure 2c), while the matrix is formed of nearly pure iron (point 3 in Figure 2c).

Table 1.
The X-ray microanalysis of cast iron/NbC gradient material

Point	Content of elements [% atomic]			
	Fe	Nb	C	Others
1	-	48,91	38,43	rest
2	-	32,31	61,95	rest
3	96,56	-	-	rest

To confirm the synthesis of niobium carbides, X-ray diffraction analysis was made; its results are shown in Figure 3. The results of the phase analysis have proved the presence of niobium carbide and alpha iron (ferrite).

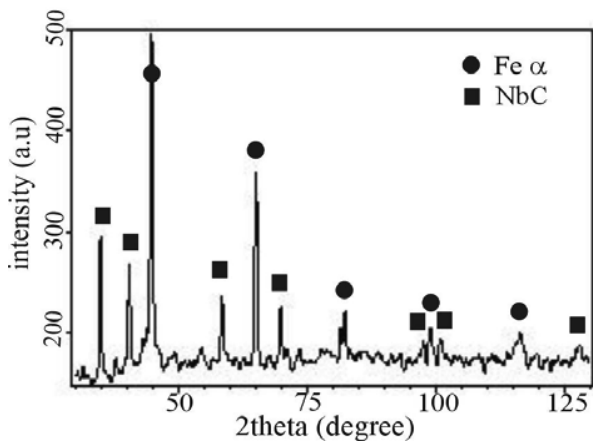
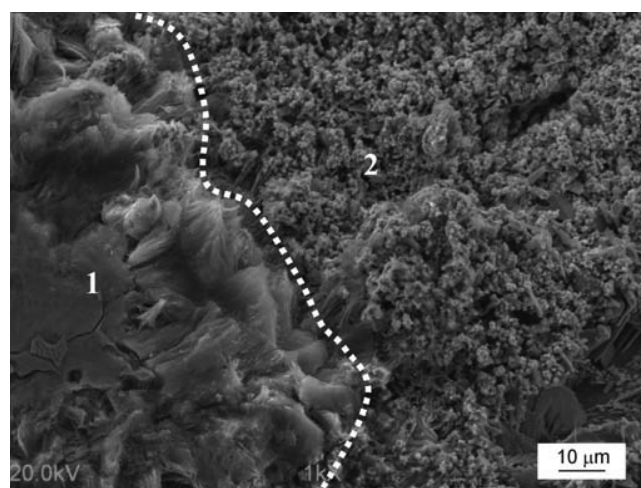


Fig.3. The phase analysis of cast iron/NbC gradient material

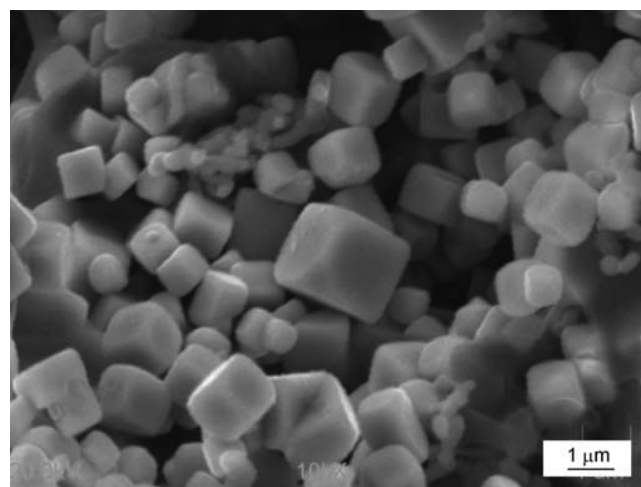
Deep etching with *aqua regia* of the subsurface layer in cast iron/NbC material has revealed the complex structure of a reinforcing layer produced in this material, characterised by high content of the niobium carbides. Broken line in Figure 4a-b marks an imaginary phase boundary between the matrix (region 1) and cast iron/NbC phases (region 2). At larger magnifications, one can see a skeleton-like structure of the NbC conglomerates, which assume the shape of the preform filled with iron alloy (Fig. 4c). This is a typical effect of the coalescence, which occurs when the time of the SHSB synthesis is short enough. Examining the crystals of niobium carbide, one can see that they are forming compact structures. Their mutual connections suggest that they have been formed during the process of crystallisation, which is best proved by Figure 5. The broken ring line in this drawing marks crystals in which some fragments of the walls have been mutually arresting their own growth. The effect was the formation of a permanent pseudo-crystalline structure.



(a)



(b)



(c)

Fig. 4. The structure of cast iron/ NbC gradient material. Specimen deep-etched with *aqua regia*

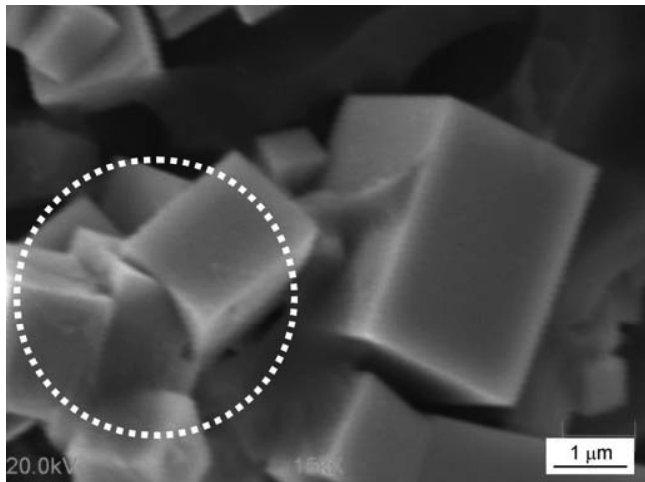


Fig. 5. The structure of cast iron/ NbC gradient material. Specimen deep-etched with *aqua regia*

Figures 6-7 shows the second produced material, in which some light-colour precipitates are visible against the grey background of the matrix.

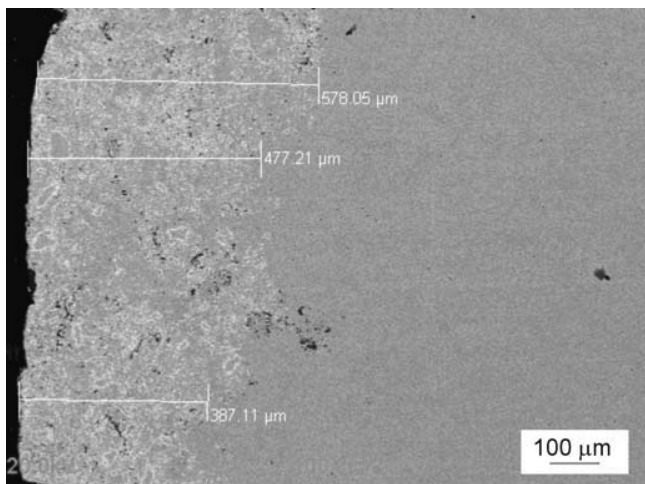


Fig. 6. The structure of Ni₃Al/NbC gradient material

The X-ray microanalysis (Tab. 2) reveals the presence of Nb and C at points 1 and 2 in Figure 7, while at points 2 and 3 the presence of Ni and Al has been detected.

Table 2.
The X-ray microanalysis of Ni₃Al/NbC gradient material

Point	Content of elements [% atomic]				
	Ni	Al	Nb	C	Others
1	-	-	20,67	76,87	rest
2	-	-	19,77	79,37	rest
3	72,12	27,44	-	-	rest
4	73,11	26,33	-	-	rest

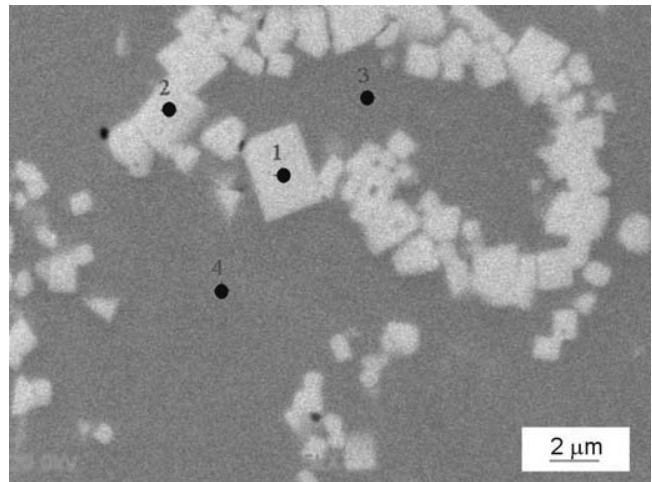


Fig. 7. The structure of Ni₃Al/NbC gradient material with marked points of X-ray microanalysis

The atomic content of the individual elements indicates the presence of the Ni₃Al and NbC phases, confirmed further by the following phase analysis (Fig.8). As shown in Figure 6, the gradient structure produced in the Ni₃Al/NbC material forms a layer 380 to 580 μm thick.

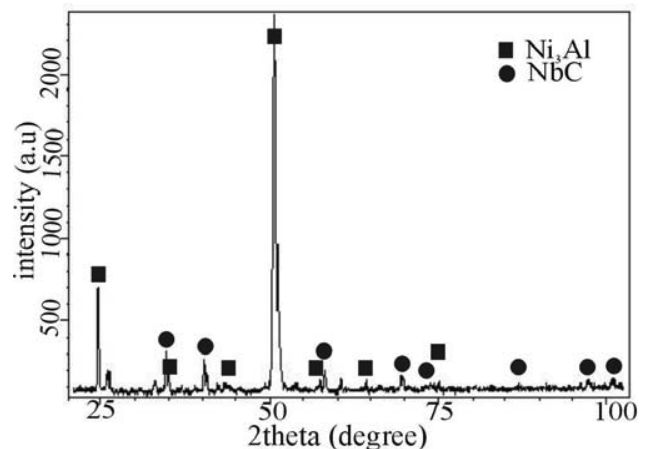


Fig. 8. The phase analysis of Ni₃Al/NbC gradient material

Figures 9-10 shows the image of a gradient structure produced in the Ni₃Al/NbC material after deep etching with *aqua regia*. One can easily note the carbide phase of even distribution present in region 1 in Figure 9a, characterised by an unstable boundary separating the carbide layer from the remaining material (region 2 in Fig. 9a).

The, generated by SHS reaction, niobium carbides form compact structures, in which one can see crystals with partly arrested planes of growth. The dimensions of the niobium carbides produced by the SHS Process in Mould (SHSM) are comprised in a range of 0,5 to 4 μm and are corresponding to the dimensions of the niobium carbides produced by the same route in the cast iron/NbC material.

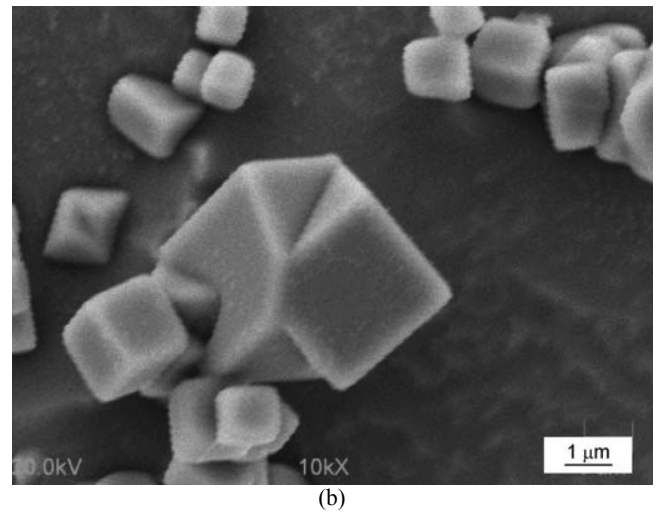
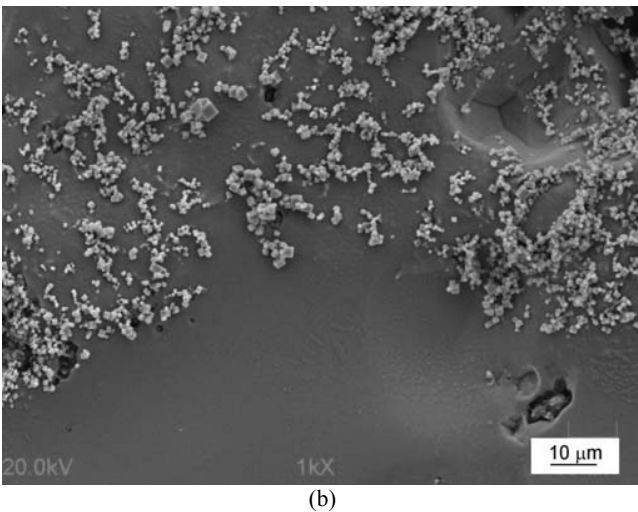
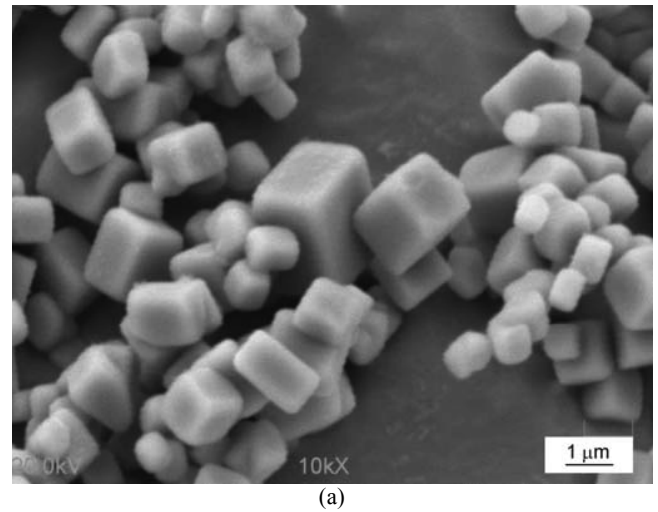
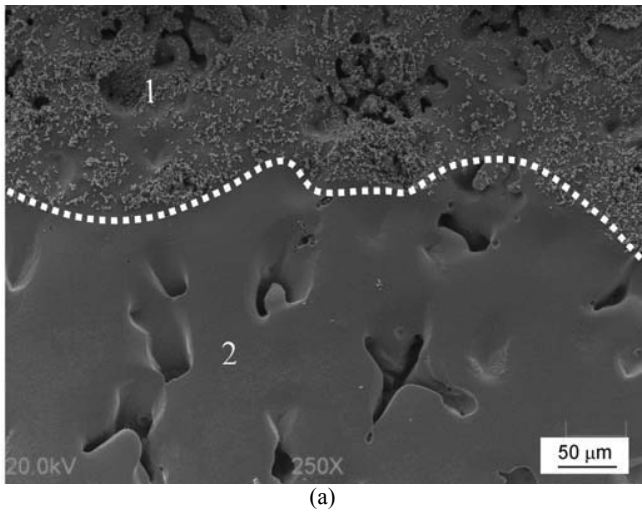


Fig. 9. The structure of $\text{Ni}_3\text{Al}/\text{NbC}$ gradient material. Specimen deep-etched with *aqua regia*

Fig. 10. The structure of $\text{Ni}_3\text{Al}/\text{NbC}$ gradient material. Specimen deep-etched with *aqua regia*

4. Discussion of results

The innovative method described in this study enables a relatively simple synthesis of the functionally gradient materials (FGMs). The fabricated materials of the cast iron/NbC (Figs. 1-5) and $\text{Ni}_3\text{Al}/\text{NbC}$ (Figs. 6-10) type are characterised by a non-homogeneous distribution of the carbide phase in matrix. The content of the reinforcing phase depends on the distance from the casting surface. The phase is generated in an SHS reaction, initiated by contact achieved between its substrates and the liquid metal at a temperature of about 1550°C , i.e. at the mould pouring temperature. By the time the metal undergoes complete solidification, this phase is diffusion spread in the subsurface layer of still liquid metal. The carbides form compact structures corresponding in their character to a skeleton preform (Figs. 4c and 10a). The size of the carbides produced by the synthesis is from 0,5 to $4\mu\text{m}$ (Fig. 4c and 10a), while the width (the range) of the subsurface layer reinforced with carbides ranges from 380 to $580\mu\text{m}$ (Fig. 6).

5. Conclusions

- A new method of the synthesis of gradient materials was developed and called SHSM (Self-Propagating High Temperature Synthesis in Mould).
- It has been stated that the said method enables synthesis of functionally gradient materials (FGMs) using different cast alloys.
- The conclusion drawn from the study is that the size of carbides formed in the SHSB process depends on the duration of the synthesis.
- The width of the reinforcing subsurface layer depends on the method used for mould coating with reactive layer.

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