EFFECT OF Ni ADDITION ON STRUCTURE FORMATION AT *IN-SITU* SYNTHESIS OF TiC HARDENED Fe-BASED POWDERED ALLOY

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Abstract: *The feachures of structure and phase formation of TiC hardened Fe-based alloy at in-situ thermal synthesis from mixtures of* TiH_2*, Fe, graphite and Ni powders had been investigated. It was shown that after synthesis at 1200* ^0C *the structure of the alloy is a skeleton of titanium carbide grains of various shapes and sizes from 1 to 20 µm cemented with Fe-based binding substance mostly located around the titanium carbide grains. The phase composition of the obtained alloy includes mainly phases of titanium carbide and α-Fe. In the case of using the initial mixture with the Ni in the composition of the alloy along with the titanium carbide solid solution of Ni in alpha-iron and Ni-based compounds were identified. When Ni is used instead of Fe in initial powder mixture it leads to a noticeable refinement of the alloy grain structure: the size of the carbide grains is generally not more than 5-7 microns. With the decrease in Ni content in the mixture and respectively increase of iron content at the same Ti and graphite content, the particle size increases markedly and approaches with 5 % of Ni to the particle size of the alloy obtained from the mixture containing no Ni.*

Keywords: ALLOY, THERMAL SYNTHESIS, MICROSTRUCTURE, GRAIN, POWDER, *TITANIUM CARBIDE, COMPOSITE*

1. Introduction

Among the groups of wear-resistant materials that made by powder metallurgy methods, TiC reinforced ferrous based composites have gained widespread use in recent years. They consist of carbides with a mass fraction from 10 to 70% and the metal bonding i.e. alloyed steel [1-7].

Titanium carbide, as the carbide component of the TiC reinforced steels, is the most often used due to a combination of properties that exceeds those of other carbides of transition metals (except tungsten carbide) [8]. Titanium carbide has a high melting point (3100 °C), Young’s modulus (451 GPa) and microhardness values up to 30 GPa. Titanium carbide has a satisfactory solubility in nickel (about 5% at 1250 °C) [9]. The low solubility of TiC in iron (about 0.5%) reduces the probability of "welding" between cutter and chip during processing steel parts, as a result titanium carbide-based alloys can be used at high cutting speeds. The advantage of using titanium carbide is also the relatively low cost of raw materials for its production. However, the main disadvantages of titanium carbide are low thermal conductivity and insufficient wettability by iron group metals during sintering.

Wetting improvement of carbide particles by a metal binder can be expected from a new technological approach for the synthesis of TiC reinforced steel powder which suggest that the carbide phase is not added into the initial powder mixture in the form of titanium carbide powder [1-4], while TiC is formed during the thermal synthesis of the alloy from powder mixtures of titanium, iron-carbon alloy and carbon powders [7, 10, 11]. Due to the presence of a low-temperature eutectic zone with a melting point of about 1085 ^oC [12] in the Fe-Ti system, interaction of Fe-Ti alloy with carbon, and high affinity of titanium to carbon at temperatures exceeding 1085 ^oC an active interaction of the components of the mixture occurs resulting into formation of titanium carbide particles [10].

In addition, J. Kubarsepp showed [2] that addition of nickel to the composition of the metal component of TiC-Fe composites reduces the wetting angle, and the maximum hardness of the alloy is observed at a nickel content of 5 to 10 %.

According to [13], nickel has a low diffusion coefficient in iron and increases the stability of austenite in powdered steels. It can be assumed that the binder in nickel-containing TiC-steel composites is most likely to contain γ-Fe based solid solutions, as well as titanium nikelide and intermetallides.

The aim of this work was to study the features of the structure and phase formation of alloys obtained by thermal synthesis from Fe-Ti-Ni-C powder mixtures.

2. Materials and experimental procedure

For experimental investigations of mixture composition influence the on the structure and phase composition of the pseudoalloys, five mixtures of iron, titanium hydride, nickel, and carbon powders were prepared (Table 1).

Table 1

Composition of the powder mixtures

Mixture number	Mixture composition, % (wt.)			
	Fe	TiH_2	C	Ni
1	20	64	16	-
2	-	64	16	20
3	5	64	16	15
4	10	64	16	10
5	15	64	16	5

The initial powders were blended in a ball mill and pressed at 600 MPa into briquettes, which were then sintered at 1200 ^oC for 1 hour in vacuum to provide the thermal synthesis of the composite.

After the thermal synthesis the samples the obtained from sponge of different compositions were cut, grinded and polished. The etching of the samples was carried out with an etchant based on a mixture of hydrofluoric and nitric acids.

Micro- and macrostructure analysis was carried out using a XJL-17 optical microscope and scanning electron microscope JEOL Supperprobe 733 with ultrasonic cleaning of fracture surface was used.

The size of titanium carbide grains was determined by the secant method on the samples studied. On each sample, at least 50 measurements were taken along arbitrarily chosen secants.

The X-Ray Diffraction (XRD) analysis of the samples was performed using DRON-3M diffractometer with CoKα radiation in the range of angles from 20 to 130 degrees. The sample during the analysis were rotated around its axis. The analysis of XRD data was performed by standard methods using the ASTM card index.

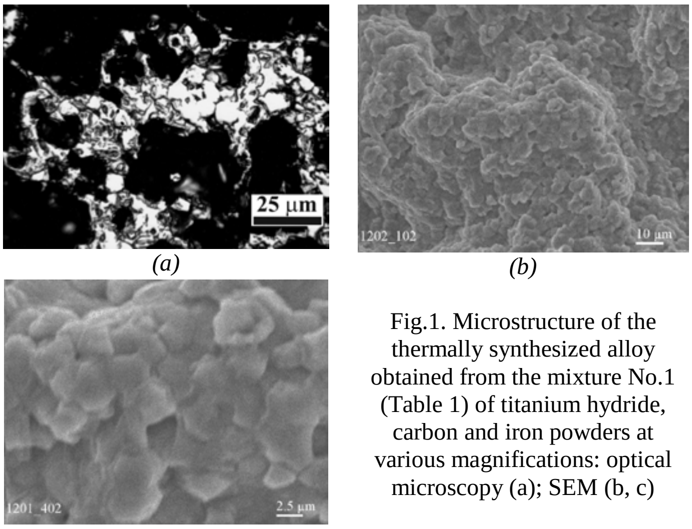
3. The results and discussion

To evaluate the influence of nickel on the structure, phase composition and properties of the synthesized alloy, the alloy obtained from the initial mixture without nickel (No. 1, Table 1) was considered as the base material.

As can be seen from Fig. 1,a,c, the structure of this alloy is a skeleton of titanium carbide grains of various shapes and sizes with a binder located predominantly around the grains of titanium carbide. Carbide grains are distributed non-homogeneously. In the optical microphoto, titanium carbide has a white or light gray color, while the solid solution is a light, formless phase and metal binder is black and rims the light grains of titanium carbide. A large portion of the sample's surface is occupied by large round pores formed mainly as shrinkage shells due to recrystallization during sintering in the presence of a liquid phase.

The structure of the alloy is not homogeneous. The size of predominantly rounded carbide grains varies from 1 μm to 20 μm (Fig. 1,c). The presence of rim zones around the grains of titanium carbide, which formed due to the interaction of carbide particles with the melt of the metal binder, is clearly observed. As the authors of [1, 2] note, the dissolution of titanium carbide in a metal binder is significantly influenced by the carbon content in carbide: the lower its content in carbide, i.e. the greater deviation from the stoichiometric composition, the higher its solubility in the steel binder.

The SEM data (Fig. 1,b,c) show that during thermal synthesis process, the partial sintering of titanium carbide powder is occurred. Melting of the particles and their mutual fusion with one another with following formation of colonies of fused carbide particles are observed too.

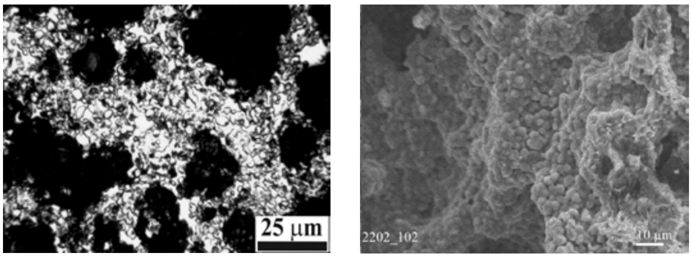


(a) *(b)*

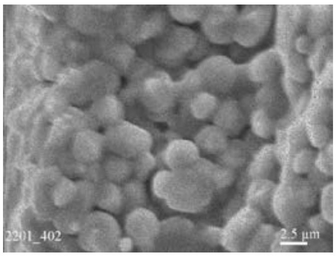
(c)

Fig.1. Microstructure of the thermally synthesized alloy obtained from the mixture No.1 (Table 1) of titanium hydride, carbon and iron powders at various magnifications: optical microscopy (a); SEM (b, c)

The replacement of iron with nickel in the initial powder mixture leads to a noticeable refinement of alloy grain structure (Fig. 2,a): the size of the carbide grains usually does not exceed 5-7 μm. It is noteworthy that the carbide skeleton becomes more disconnected (the carbide grains act as separate islands) (Fig. 2,b,c) in comparison with the alloy obtained from the mixture No.1 with iron as the metal binder.



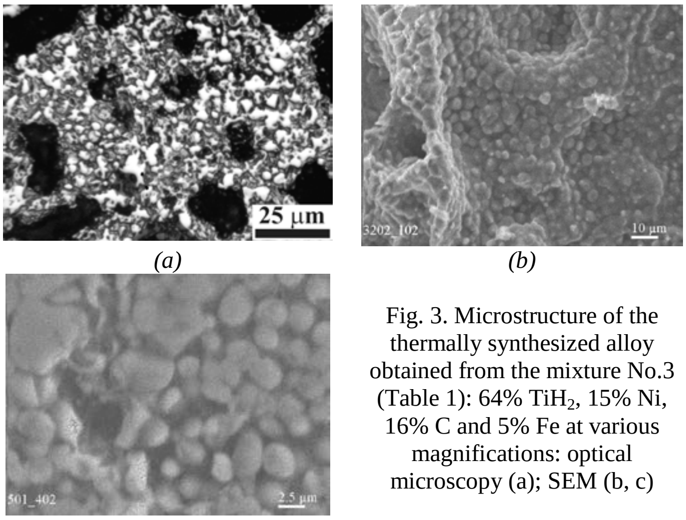
(a) *(b)*



(c)

Fig. 2. Microstructure of the thermally synthesized alloy obtained from the mixture No.2 (Table 1) of titanium hydride, carbon and nickel powders at various magnifications: optical microscopy (a); SEM (b, c)

In case of use of Fe-Ni alloy as the metallic binder of the composite with iron composition in the initial mixture of 5÷15% (mixtures No.3 and 4) a slight increase in size of carbide grains and somewhat larger grain size distribution can be noted in comparison with Fe-free alloy No.2. Internal fragmentation of carbide grains is observed too. Along with fine carbides of 1÷2 μm in size, the carbide grains are combined into large colonies with the size more than 25 μm (fig.3).

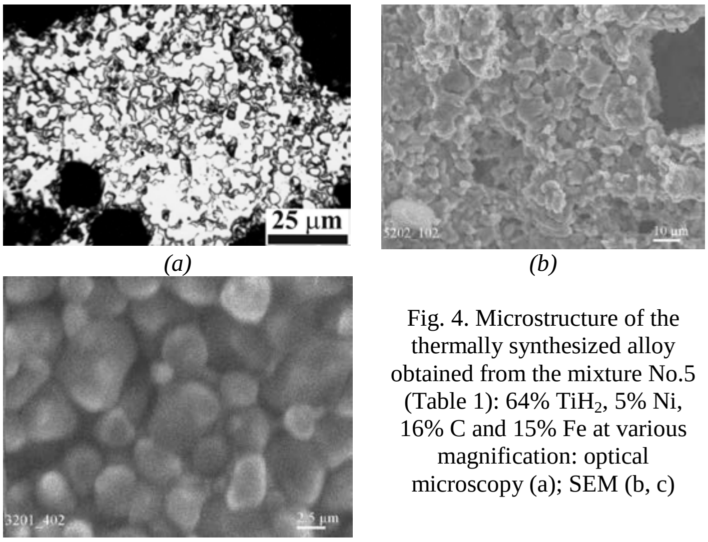


(a) *(b)*

(c)

Fig. 3. Microstructure of the thermally synthesized alloy obtained from the mixture No.3 (Table 1): 64% TiH_2, 15% Ni, 16% C and 5% Fe at various magnifications: optical microscopy (a); SEM (b, c)

As the content of nickel in the mixture decreases to 5%, the size of the particles (mixture No.5) increases noticeably (Fig. 4) and approaches the size of the particles of the alloy, obtained from the mixture No.1 without nickel.



(a) *(b)*

(c)

Fig. 4. Microstructure of the thermally synthesized alloy obtained from the mixture No.5 (Table 1): 64% TiH_2, 5% Ni, 16% C and 15% Fe at various magnification: optical microscopy (a); SEM (b, c)

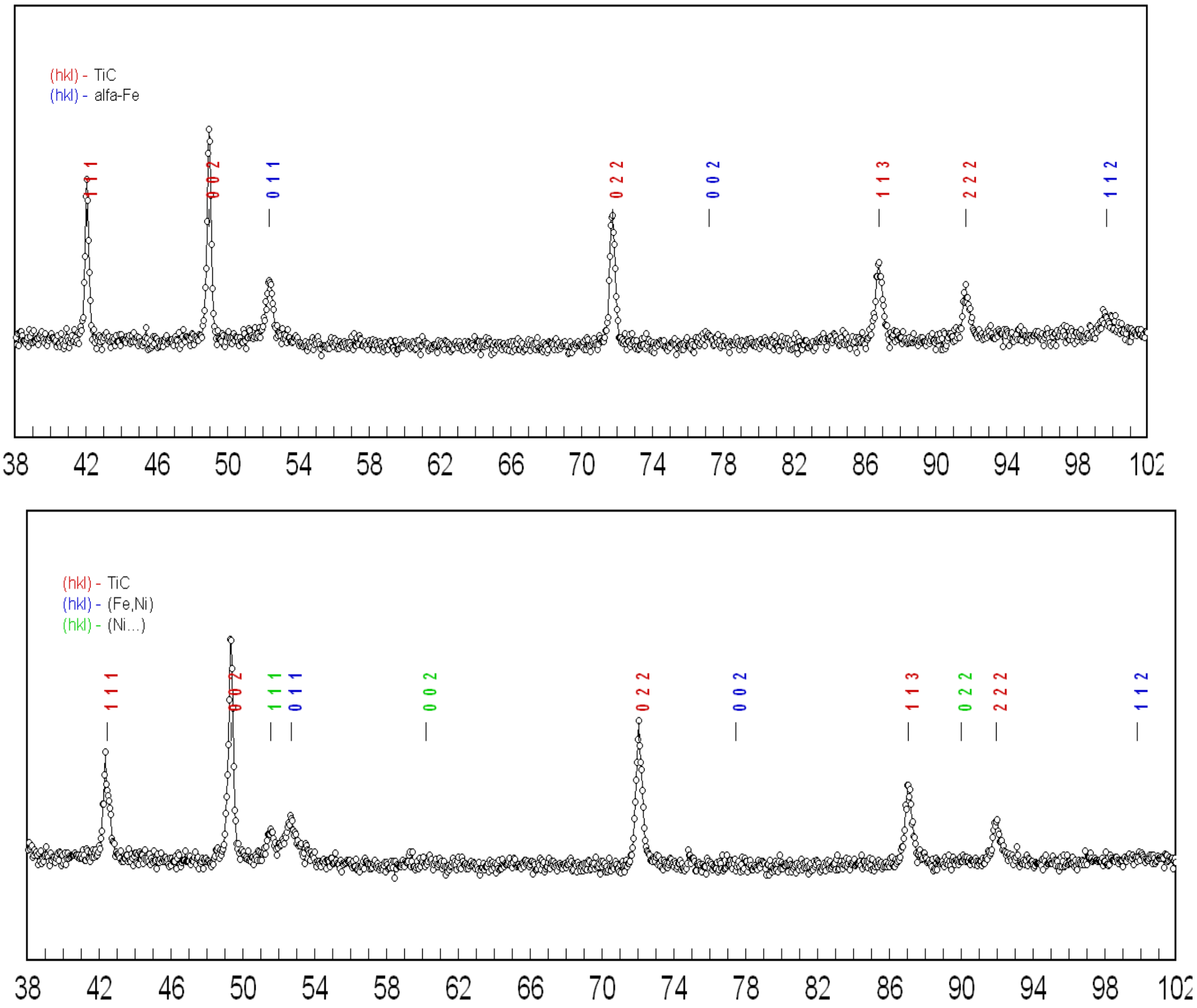


Fig. 5. XRD patterns of samples obtained from mixtures Fe-Ti-C (No.1) (a) and Fe-Ti-Ni-C (No.5) (b)

The XRD analysis of the sintered composites showed that in case of the alloy synthesized from the mixture of Fe, TiH_2 and C powders (mixture No. 1), the thermally synthesized alloy consists of TiC and solid solution of α-Fe (Fig. 5a). When the initial mixture with nickel is used (mixture No. 5), the solid solution of nickel in alpha-iron and nickel compounds with a lattice period of a = 4.3198 A is observed along with the titanium carbide phase.

According to the Wulf-Bragg relation, the lattice period of titanium carbide in the obtained alloys is a = 4.3266 A for cubic syngony (Fm3m), which corresponds to titanium carbide with $TiC_{0.82}$ stoichiometry [8]. The size of the coherent scattering blocks for titanium carbide in the sample obtained from the mixture No. 5 is 217 A, which is almost 20% less than that for the sample sintered from the mixture No.1 without nickel (259 A). It seems that these are small titanium carbides (0.2÷0.3 μm) in the alloy, which are indistinguishable for optical microscopy but act as single crystallites for an X-ray radiation by scattering it coherently.

The average grain size of titanium carbide grains are in the range of 0.5 μm to 5 μm for all samples as was defined from the micrographs by the secant method (Fig. 6,a). At the same time, the highest amount of fine grains with sizes of 1÷2 μm was observed for alloys obtained from mixtures No.2 and 3 with high nickel content (20 and 15%, respectively), whereas grains of 8 μm to 17 μm in size were observed only in alloys obtained from nickel-free mixture (No.1) and mixture No.5 with a minimum (5%) Ni content.

The dependence of the minimum and maximum carbide grains size of on the nickel content in the initial mixture is also of interest. According to Fig. 6,b, the minimum grain size is 0.5÷1.0 μm, regardless of the nickel content in the mixture, while the maximum grain size (~17 μm) is substantially higher for nickel-free and low-nickel alloys (mixtures No.1 and 5). Whereas an increase of Ni content in the mixture to 10÷20% results in significant reduction of the maximum grain size to 6÷7 μm, which confirms the conclusion about the dispersive role of nickel additives.

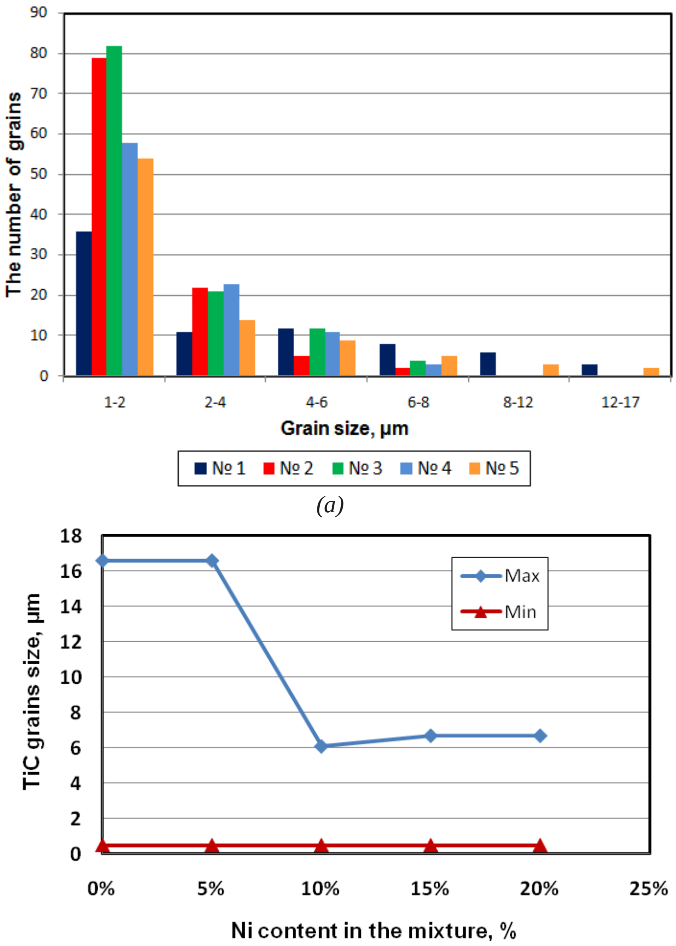
Conclusions

1) It is shown that thermal synthesis is an efficient method for obtaining composite powders of TiC hardened steels from mixtures of iron, titanium hydride and graphite at relatively low temperatures and can be used for deposition of wear-resistant coatings and fabrication of bulk parts by powder metallurgy methods.

2) The structure of the synthesized alloy is a porous skeleton made of titanium carbide grains of various shapes and sizes and binder located predominantly around titanium carbide grains.

3) The phase composition of the alloy synthesized from the mixture of Fe, TiH_2 and graphite powders consists of titanium carbide and solid solution of α-iron, whereas in the case of using the initial mixture with nickel, a solid solution of nickel in α-iron and nickel compounds with a lattice period of a = 4.3198 A is observed along with titanium carbide.

4) It is shown that the addition of 10÷20% Ni to the initial mixture leads to significant dispersion of the carbide phase (up to 1÷6 μm) in the synthesized alloy, whereas the maximum grain size of carbide phase reaches 17÷20 μm for nickel-free and low-nickel alloys.



(a)

(b)

Fig. 6. The relation of TiC grains size distribution of powder mixture content (a) and of Ni content in the mixture (b)

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