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Features of the structure formation of sintered powder materials using waste metal processing of steel workpieces

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ABSTRACT

Introduction. Manufacturing processes, to one degree or another, are associated with the metal waste production in the form of metal chips. The development of technologies for recycling of waste from mechanical facilities is a popular solution both from the resource saving and from an environmental points of view. Among many traditional approaches to the problem of recycling metal chips, the most interesting may be the method of using chips as one of the components in a powder material. **The aim of this work** is to analyze the possibility of using metalworking wastes from steel 45 (metal chips) in powder compositions based on titanium and aluminum not only as a source of iron, but also as a possible source of Fe_2O_3 oxide. Attention to the oxide was paid in terms of initiating reduction reactions in the powder mixture based on titanium and aluminum with the formation of the Al_2O_3 oxide phase to obtain a metal matrix composite. **Research methods**: steel chips after processing workpieces from steel 45 were additionally oxidized in water and crushed in a vibrating mill to an average particle size of 300 μm for use in powder compositions with titanium and aluminum powders. Grinded and oxidized chips were mixed with titanium and aluminum powders in various proportions in order to study its interaction with these powder components. The obtained mixtures were pressed in the form of cylindrical samples and sintered in a vacuum furnace at a temperature of 1,000 °C. The phase composition and microstructure were studied using an *XRD-6000* X-ray diffractometer with CuKα – radiation and an *AXIOVERT-200MAT* optical microscope. **Results and discussions**. It is shown that after milling without coolant, steel 45 chips did not accumulate a noticeable amount of iron oxides, which required additional oxidizing procedures. The interaction of grinded oxidized chips with the components of powder mixtures is considered, and its effect on volumetric changes in compacts and structure formation of metal-matrix composites is shown. The results of optical metallography and X-ray diffraction analysis (*XRD*) of sintered powder compositions using oxidized ground chips of steel 45 made it possible to evaluate the ongoing processes of structure formation depending on the combination of interacting components, its mutual influence, and the prospects for obtaining composites with a dispersed oxide phase.

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Introduction

The production processes of mechanical engineering companies are associated with the formation of a large variety of waste that may include valuable secondary raw materials. Solutions related to technologies of processing and recycling waste from the engineering production and its inclusion into the technological cycle are widely in demand among other problems of resource saving and reducing the environmental load.

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This topic is relevant to conditions of rational use of natural resources, especially metals and alloys, widely used in mechanical engineering. Metalworking using various machines makes the greatest contribution to the total waste of the production chains in mechanical engineering. Metal chip is always formed in the manufacturing process of any metal part regardless of the processing type and the used tool. Efficient disposal of the metal chip after machining is a serious problem for mechanical engineering companies, because its state is very different from the initial state of the workpiece. Traditionally, metal chip is polluted with all sorts of impurities in every type of metalworking. Besides the cutting fluid (coolant), there may be oil, moisture, sand particles, sludge, and other debris. All this makes it difficult to recycle and process it directly in the production workshop. Rust is another problem in the metal chip recycling. It starts to form intensively immediately after the processing and continues to grow with the time of waste storage. Mechanical engineering companies most often accumulate metal waste and transfer it to metallurgical production for remelting in order to avoid all these problems [1–5]. In this regard, the possibility of compacting metal chip waste is the priority task for the metal chip utilization in order to minimize the volume and facilitate transportation for further remelting [3]. A number of works [6–8] propose to consider the production waste as an independent resource in the form of modified blend for further use as semifinished items. Some of the most common materials for mechanical engineering are various grades of steel. Respectively, a significant amount of waste will be the steel chip. On the other hand, the steel chip can be used as a resource not only for secondary melting, but also as a source of components for powder technology. Firstly, the chip, regardless of the alloy, is a material with the defective structure that was formed as a result of cutting [2, 7]. It can contribute to its dispersing and the application of hot densification technologies of the already powdered product.

Secondly, the chip is a sufficiently activated material which can be further grinded and oxidized. Thirdly, the great importance has the influence of the processing medium with using coolant, the accompanying oxidation processes, etc. [5]. This all makes the steel chip a convenient raw material for the preparation of powder compositions with a specific combination of components.

The steel chip can also be interesting not only as a source of iron, but also as an oxide-containing component for obtaining composite materials with oxide inclusions. The use of oxides in composite materials science has been developing for decades [9–15]. The combination “oxide – metal base” depends on the purpose and operating conditions of products made from this composite. In this case, one can consider not only bulk materials, but also surfaces modified by composite coatings [16].

If we consider the steel chip as a potential source of oxide inclusions, then the analysis of metal components that can be used in the composition with the grinded oxidized steel chip plays an important role. Titanium- and aluminum-based powder materials are the most interesting group of metal components that can be considered as matrix material when using the recycled metal chip. In particular, studies of composite materials based on titanium with various refractory additives from compounds of carbides, nitrides, borides, silicides, and oxides are well known [9–10, 14]. Composites based on an aluminum matrix with addition of refractory compounds are also of interest [13, 17, 18]. The group of composites based on the *Ti–Al* system, which can be considered as a matrix of composite material, does not lose its relevance in the research [13, 19–22]. A wide range of technological processes related both to various types of cladding and surface modifications [19] and to the processes of *SHS*, electrospark sintering and other types of consolidation of powder components are used to obtain metal-matrix composites [9–16, 18–23]. Among these methods, a vacuum sintering with the controlled heating seems to be a fairly simple and convenient option for studying the physical and chemical processes that can occur in complex systems with interacting components, including oxide compounds. The vacuum sintering is convenient to use at the initial stage of the study, because it is difficult to predict the possible diffusion-reaction processes that can occur in the mixture under study with products from metal processing waste during other technological processes for obtaining powder composites (*SHS*, electrospark and laser sintering, thermal explosion, etc.).

Predicting the behavior is an extremely difficult task for the materials based on titanium and aluminum with the addition of the steel chip which in turn is iron with the addition of other impurities in various proportions. With a general approach, one has to rely on the known data of the basic systems of *Ti–Al*, *Ti–Fe*, *Al–Fe*, and also *Ti–Al–Fe–O*, taking into account the oxidation of the chip [20–22, 24–25]. In this regard, it is relevant to study the powder products synthesized under vacuum sintering conditions due to the interaction of matrix components (*Ti*, *Al*) with the treated steel chip. Thus, **the aim of this study** is an analysis of the structural-phase state of sintered powder products with different combinations of matrix components when using the grinded steel chip. An evaluation of the interaction of the treated steel chip with titanium and aluminum powder components will allow determining possible research areas for achieving acceptable properties in terms of compositions control, processing modes, and consolidation conditions.

Materials and methods

For experimental research, the powder mixtures were prepared using both industrial powders of titanium (*TPP-8* with a dispersion of < 125–160 μm), aluminum (*PA-4*, < 100 μm), and a powder from grinded and sieved to 300 μm, additionally oxidized *steel 45* chip. The initial state of the chip is shown in Fig. 1. Waste from milling workpieces made of *steel 45* without using cutting fluids was used for the research. It was assumed that a significant part of the oxide film would be formed on the chip as a result of this technological operation. The X-ray diffraction analysis of the *steel 45* chip showed an almost standard set of characteristic phases of this steel. Consequently, no noticeable number of oxides (10–15 vol. % Fe_2O_3) was formed (Fig. 2, *a*).

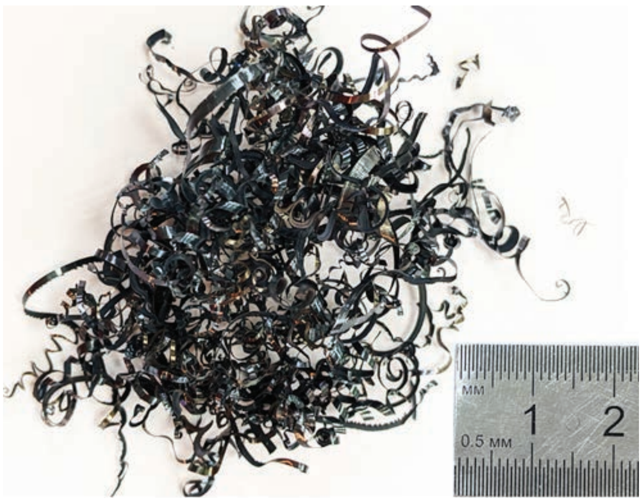
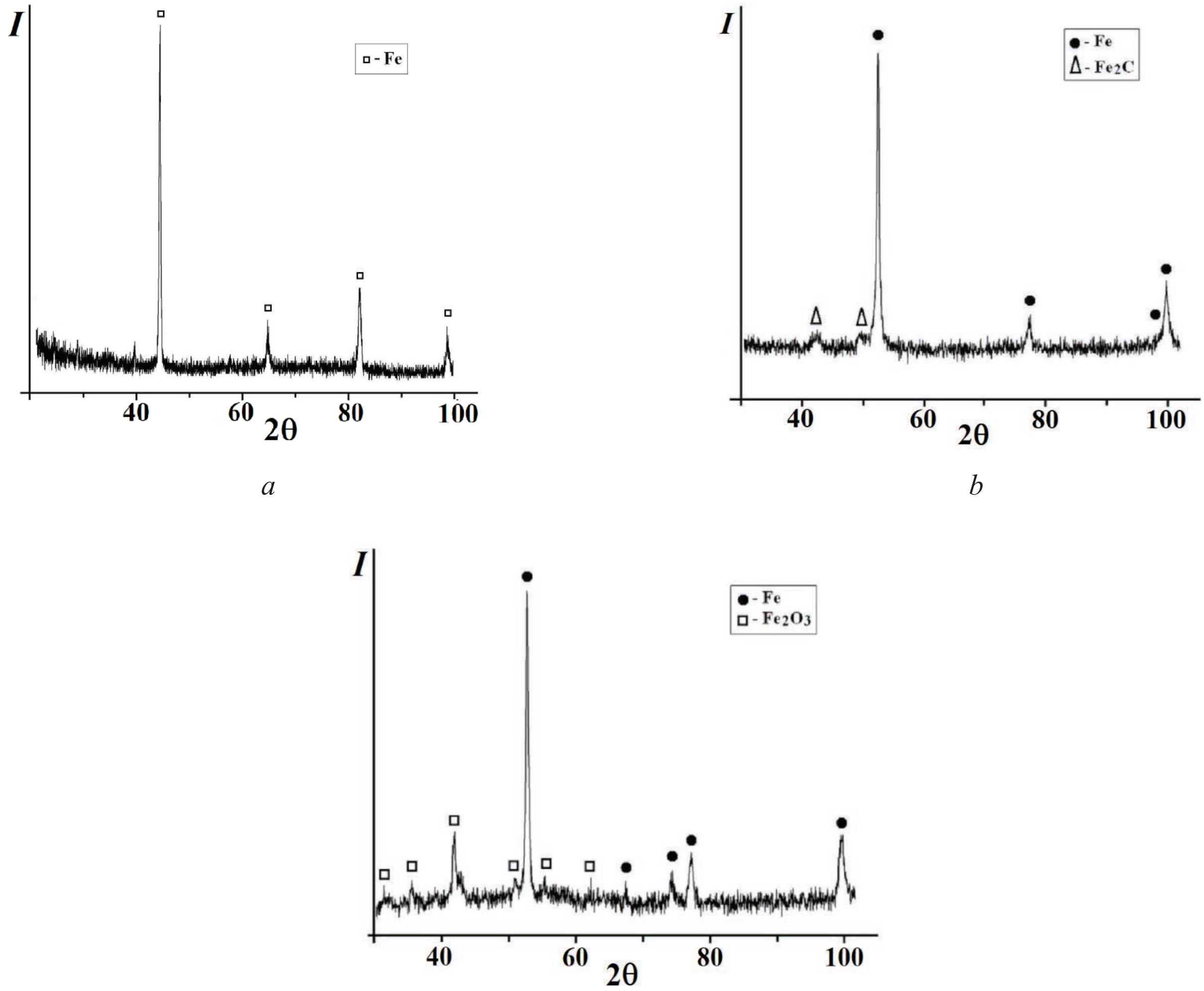


Fig. 1. Chip appearance after milling a *steel 45* workpiece

It is obvious that an increased oxygen content is present, but it is distributed in the form of local small (possibly nanoscale) oxide inclusions that are beyond the sensitivity of an X-ray diffractometer. In this regard, it was decided to oxidize further the chip using the simplest and most accessible way, such as keeping the chip in water for 48 hours. As another method of oxidation, a prevalent method of air annealing in a muffle furnace was used (up to 400 °C). However, the Fe_2C precipitation (Fig. 2, *b*) occurred in the chip after such heat treatment. Therefore, the preference was given to the oxidation in water. As a result, the Fe_2O_3 phase was formed in sufficient volume (Fig. 2, *c*).

Grinding of small chip pieces was carried out using a vibrating mill in the presence of steel balls in a ratio of 20:1 (balls/chip). Such treatment made it possible to stimulate further the formation of iron oxides. The chip was grinded into various fractions as the result of vibration grinding. After that, particles up to 300 μm have been sieved out. Smaller fractions were not sieved out, since its output was less than 10 % of the processed chip volume. The compositions of the mixtures used provided several options of combining components: *Al* + chip (*steel 45*); *Ti* + chip (*steel 45*) and *Ti* + *Al* + chip (*steel 45*). The components ratio in the mixtures was determined based on possible interaction reactions. For the first option (*Al* + chip (*steel 45*)), the number of interacting components should be enough to initiate the iron reduction reactions from the Fe_2O_3 iron oxide formed in the steel chip. For the second option, the powder fraction of the processed steel chip in titanium corresponded to an area comparable with the limiting solubility of iron in titanium [25]. The third option of the mixture corresponded to the composition, in which the selected ratio of components could stimulate both reduction reactions (metallothermy) and synthesis of intermetallics. The powder mixtures used in the experiments are presented in Table 1.

The powders were mixed in an axial mixer for 4 hours. The obtained mixtures were pressed using a cylindrical mold with a floating punch to obtain samples with 10–15 mm in height and 10 mm in diameter. The studied samples with an initial porosity of 25–30 % were sintered in a vacuum furnace at a temperature



a *b*

c

Fig. 2. Phase composition of metal chips from steel 45:
a – initial state; *b* – after annealing in a furnace at 400 °C in air; *c* – after holding in water for 48 hours and drying at room temperature

Table 1

The composition of the studied powders with recycled steel 45 chips

No.	Composition	Components, wt.%		
		Steel 45 chip powder	*Al*	*Ti*
1	*Al* + chips (*steel 45*)	75	25	-
2	*Ti* + chips (*steel 45*)	25	–	75
3	*Ti* + *Al* + chips (*steel 45*)	23	8	69

of 1,000 °C with an exposure time of 60 minutes and a heating rate in the range of 5–10 °/min. The standard expression was used to determine the porosity:

$$\Theta = 100\left(1 - \frac{\rho_{real}}{\rho_{theor}}\right), \tag{1}$$

where θ – porosity, %; ρ_{real} – the actual sample density; ρ_{theor} – the theoretical density of the powder mixture calculated by the additive method, where the initial components data was used in the calculation before sintering, and after sintering – qualitative and quantitative data of the *XRD*.

Volumetric changes and transformation of the pore structure are additional indirect indicators of structural and phase changes in powder materials. Thus, volume changes were calculated for the samples of those powder mixtures compositions that retained its shape. Volume changes were defined as the relative change in the samples volume before and after sintering:

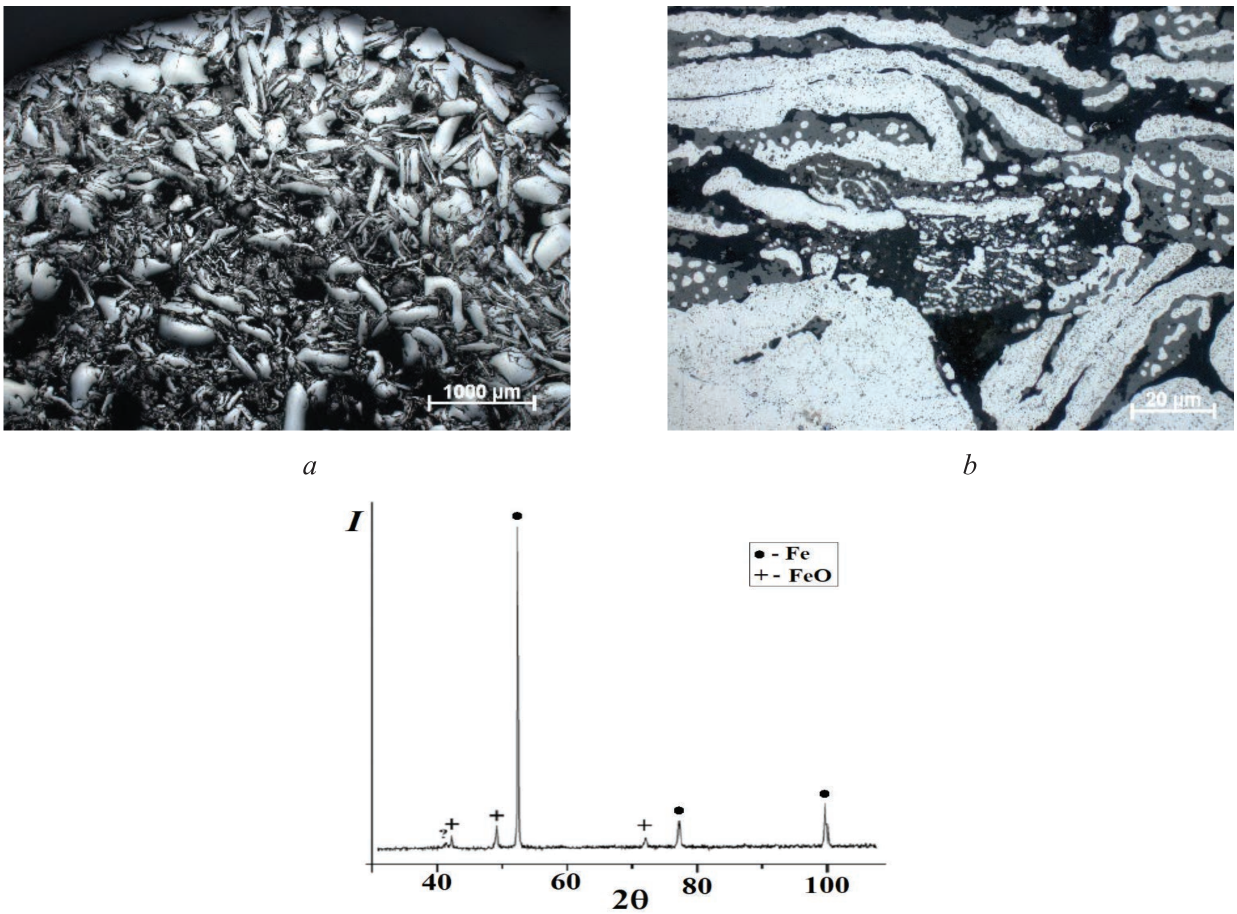
$$\frac{\Delta V}{V_0} = \left(\frac{V_0 - V}{V_0}\right), \% \qquad (2)$$

where V_0. – the initial sample volume; V – the sample volume after sintering.

Structural studies were carried out using optical microscopy, X-ray diffraction analysis and energy dispersive microanalysis (*AXIOVERT-200MAT* optical microscope, *Shimadzu XRD–6000* X-ray diffractometer, CuK_α radiation, *TESCAN MIRA 3LMU* scanning electron microscope). The phase composition was analyzed using the *PDF 4+* databases, as well as the *POWDER CELL 2.4* full-profile analysis program using the *Rietveld* phase quantification.

Results and discussions

Control compacts were prepared from the powder of grinded oxidized chip and sintered along with the samples of other compositions investigated to understand the behavior of treated chip from *steel 45* during sintering. The general view, morphological features and detected phases of the sintered processed chip are shown in Fig. 3. The sintered chip microstructure shows the specific shape of fragmented steel particles, where small oxide inclusions are observed (Fig. 3, *b*). X-ray diffraction analysis of the sintered treated chip showed the degradation of the initial oxide Fe_2O_3 with the transition to FeO monoxide (Fig. 3, *c*).



a *b* *c*

Fig. 3. General view (*a*) of a sintered compact made of processed chips, its microstructure (*b*) and phase composition (*c*)

Aluminum was added to the *steel 45* chip according to the assumption that this mixture is an active-interacting *Fe–Al–O* composition. In this case, several parallel-sequential reactions can be initiated, including a reduction reaction. As it is known, the formation of several intermetallic compounds (Fe_3Al, $FeAl$, $FeAl_2$, Fe_2Al_5 и $FeAl_3$) is possible in the *Fe–Al* system according to the equilibrium state diagram [25]. The reduction reaction is not excluded if the Fe_2O_3 iron oxide is presented in the treated chip: $Fe_2O_3 + Al \rightarrow Al_2O_3 + Fe$. Both the formation of intermetallic compounds and aluminothermy are exothermic reactions, which can significantly increase the current heating temperature. In this case, the sintering process can turn into a thermal explosion. Aluminum interacts with grinded particles of oxidized *steel 45* chip under the condition of a new powder product synthesis with a complex phase composition (Fig. 4). This powder material can be considered as a resource for various additive manufacturing technologies or as a precursor in other powder mixtures.

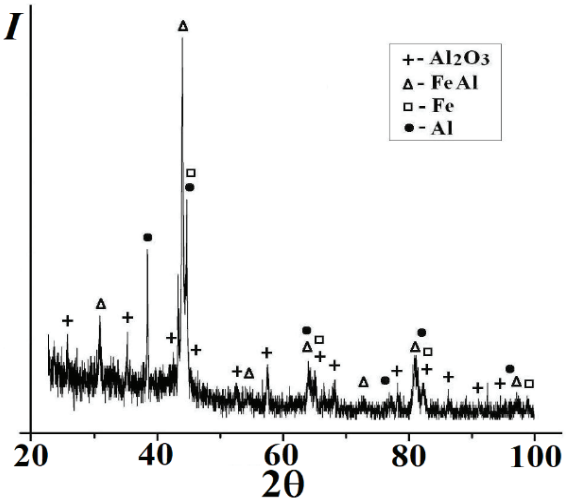
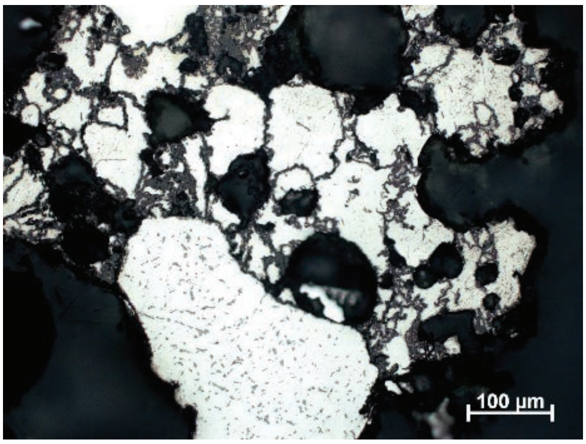


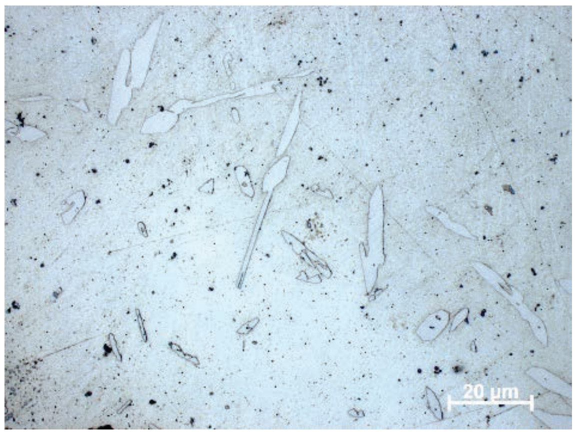
Fig. 4. Phase composition of the sintered powder product from *Al* + chips (*steel 45*) mixture

At first glance, it can be assumed that the basis of the synthesized particles from a mixture of aluminum and the treated chip is a metal matrix with needle-like inclusions of iron aluminides and inclusions of the Al_2O_3 oxide phase (Fig. 5). The calculation of the phase ratio in the synthesized product showed that the significant part of the volume belonged to the *FeAl* (up to 30 vol. %) and Al_2O_3 (up to 17 vol. %) phases. The *XRD* also detects free iron (23 vol. %) and aluminum (15 vol. %). According to the main chemical element distribution map (Fig. 6), iron and oxygen are distributed within the volume of the synthesized particles. Aluminum is also present there, but in combination with iron (*FeAl*) or in the form of Al_2O_3 oxide. Areas with predominant iron content prevail directly in the volume of particles, while free aluminum is concentrated at their periphery (Fig. 6, c).

The interaction of the treated steel chip with titanium was considered using the example of the *Ti* + chips (*steel 45*) composition, where titanium was the basis, and the chip was an alloying additive, unlike the previous mixture. In this case, no extreme reactions were expected; the process was carried out under typical solid phase sintering conditions. The chosen sintering temperature (1,000 °C) is low for this composition, while a possible liquid phase (eutectic) in the *Ti–Fe* system is formed at 1,085 °C according to the equilibrium state diagram. Therefore, a titanium phase is predominantly observed with a small inclusion of free

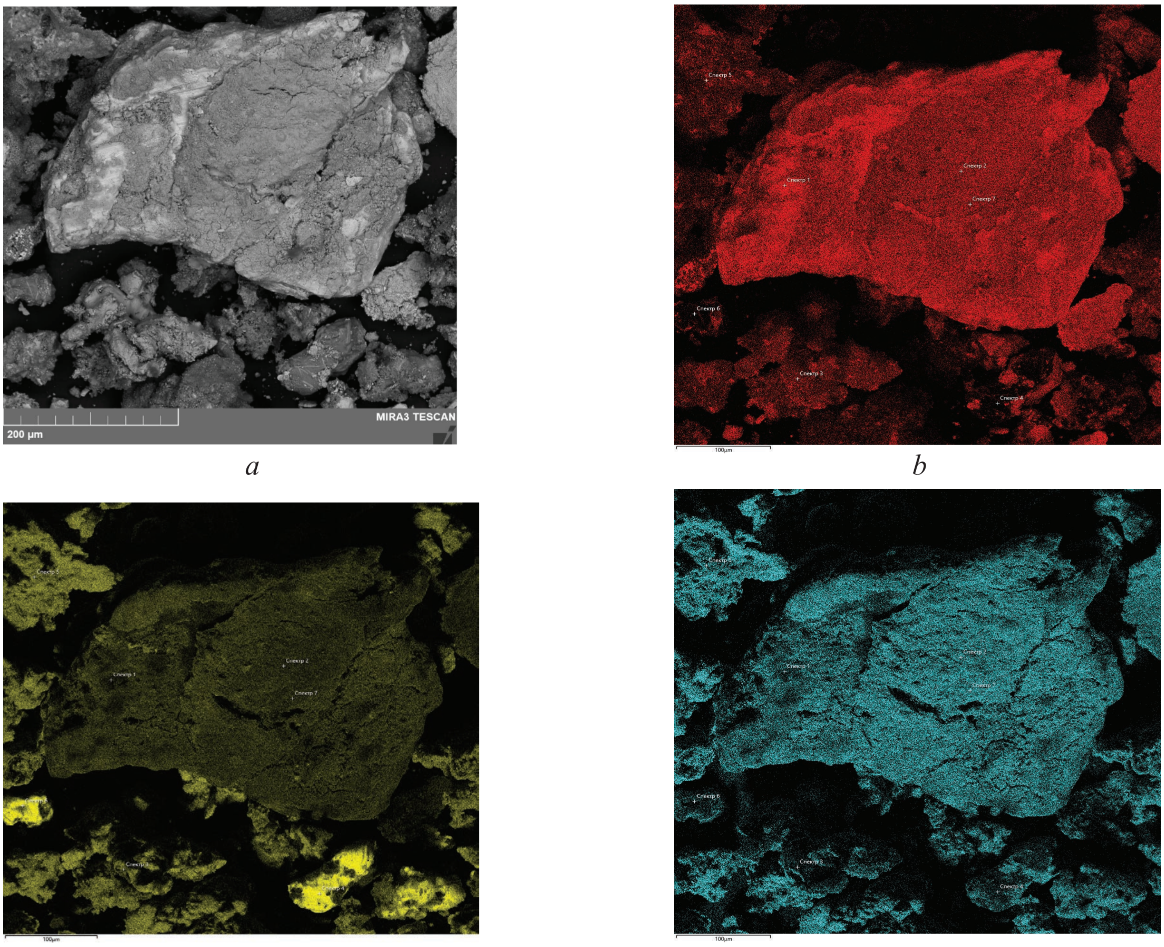


a



b

Fig. 5. Microstructure of the powder product obtained by sintering a mixture of *Al* + chips (*steel 45*): the general grain view of powder particles (*a*) and the internal structure (*b*)



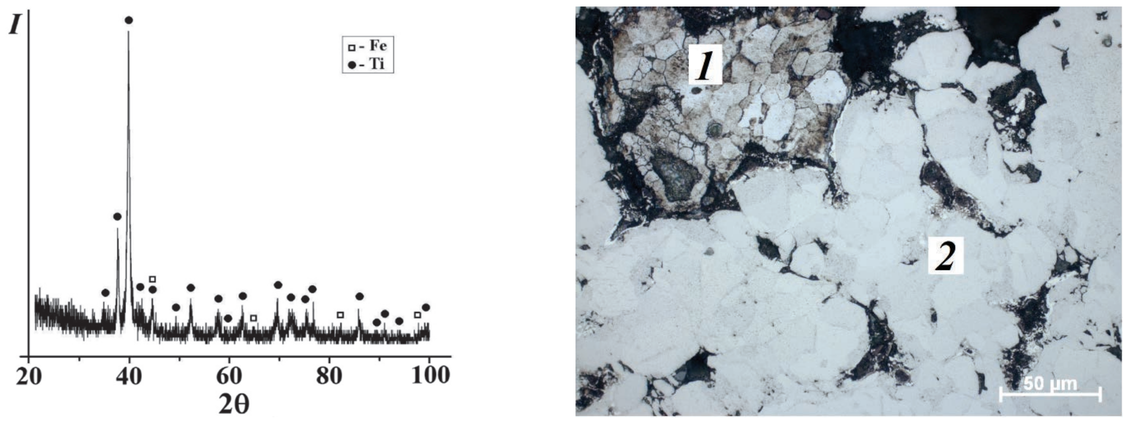
a *b*

c *d*

Fig. 6. Chemical elements distribution in the sintered powder product of the *Al* + chips (*steel 45*) mixture:
SEM secondary electron image (*a*); in the characteristic radiation *Fe* (*b*); *Al* (*c*) and *O* (*d*)

iron as a result of compacts sintering from the *Ti* + chips (*steel 45*) mixture (Fig. 7). The general picture of the sintered material microstructure corresponds to the configuration of the titanium matrix with inclusions of iron residues (Fig. 7, *b*).

Oxygen, which was initially in oxide inclusions on the particles of the treated steel chip, most likely migrated into the titanium matrix. Perhaps, some of the iron also diffused. The presence of oxygen up



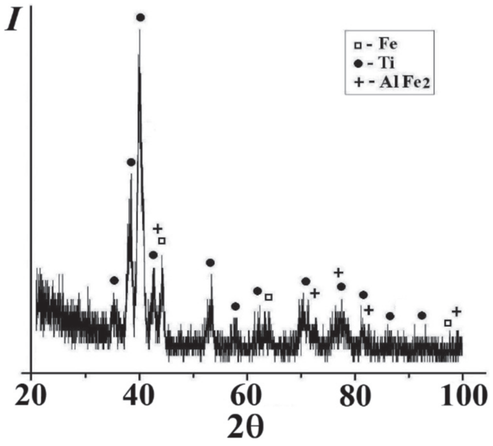
a *b*

Fig. 7. Phase composition (*a*) and microstructure (*b*) of sintered compacts from *Ti* + chips (*steel 45*) mixture; *1* – an area rich in free iron; *2* – solid solution area based on *α-Ti*

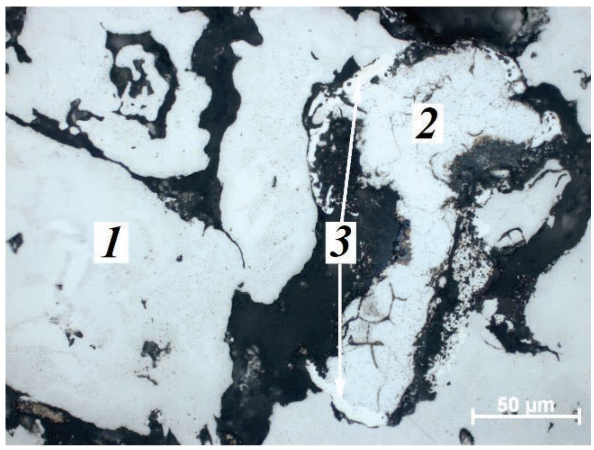
to 2.5 % in sintered samples is confirmed by the data of elemental analysis of gaseous impurities, performed using the *LECO ONH-836* analyzer. The results of X-ray diffraction analysis of sintered samples of the *Ti* + chips (*steel 45*) composition make it possible to identify the titanium base as a non-equilibrium solid solution based on *α-Ti*, the fraction of which reaches 91 vol. %. The remainder is free iron (9 vol. %) at the locations of fragments particles of the steel chip.

The variant, when during the sintering process the powdered oxidized chip simultaneously interacts with titanium and aluminum, is shown in Fig. 8. The ratio of components admits both the cross-synthesis initiation of intermetallic compounds and the iron reduction reaction from oxide inclusions on steel chip particles. The actual phase composition (Fig. 8, *a*) shows that in this case the large volume (about 67 vol. %) of the non-equilibrium phase of the solid solution based on *α–Ti* is formed, where both a part of aluminum and oxygen can diffuse. The formation of another non-equilibrium phase up to 12 vol. %, the stoichiometry of which is close to $AlFe_2$ (according to the *PDF 4+* database file cabinet), can occur at the boundary of steel chip particles upon contact with aluminum particles. Also, in the sintered sample from this composition, X-ray diffraction analysis determined the iron content up to 21 vol. %, perhaps some of it is the reduction reaction product from the formed oxide phases on the grinded oxidized steel chip when interacting with aluminum. The oxide phases were not explicitly determined by X-ray diffraction analysis despite the oxygen presence, which was identified by the analyzer at a level of 1.8 %. It is obvious that the selected ratio of components (*Ti*, *Al* and grinded *steel 45* chip) and the oxidation degree of the chip (not more than 30 vol. % Fe_2O_3) did not provide the required amount of reduction reaction products ($Fe_2O_3 + Al \rightarrow Al_2O_3 + Fe$).

Considering the variants of compositions presented in the work using the treated steel chip, it can be unequivocally stated that the grinded oxidized chip is an active interacting component in the studied compositions. The oxide phase presence on fragmented chip particles not only doesn't prevent the reaction-diffusion interaction with other components, but also promotes the implementation of additional reaction processes. As an indirect confirmation of such processes, one can use the results of the analysis of volumetric changes in compacts after sintering. Table 2 shows the changes in the volumes of sintered compacts, with the exception of the mixture with aluminum (*Al* + chips (*steel 45*)) which has lost its initial shape due to an intense exothermic reaction. Negative values show volumetric growth of compacts due to the formation of new phases, migration of elements from one group of components to another, formation of pores in place of molten aluminum which, in turn, migrated to other components, increasing the volume of their grains, etc. The most notable volume increase of compacts is observed after sintering of the last variant of the *Ti* + *Al* + chips (*steel 45*) mixture. Here, several diffusion processes associated with aluminum migration



a



b

Fig. 8. Phase composition (*a*) and microstructure (*b*) of sintered specimens from *Ti* + *Al* + chips (*steel 45*) mixture; *1* – solid solution area based on *α-Ti*; *2* – area rich in free iron; *3* – area corresponding to the nonequilibrium $AlFe_2$ phase

Table 2

Volumetric changes of the sintered compacts with recycled steel 45 chips, %

No.	Composition	$\Delta V/V_0$, %
1	*Steel 45* chip powder	6.5
2	*Ti* + chips (*steel 45*)	–2.7
3	*Ti* + *Al* + chips (*steel 45*)	–26.7

appear at once that allow not only increasing the titanium lattice volume, but also affect the dimensional parameters of iron. For comparison, Table 2 shows the volumetric changes after sintering in compacts made from the treated chip without the addition of other components (aluminum and titanium), which demonstrate the standard shrinkage for this case.

Conclusions

After simple available additional operations of oxidation and grinding, metalworking wastes from *steel 45* can be used in powder technologies not only as sources of iron, but also of its oxides.

An acceptable powder component-adding can be obtained for further use in multicomponent mixtures for the synthesis of metal-matrix composites based on titanium and aluminum using a simple method of steel chip preparing (treatment). The steel chip is well grinded after additional oxidation in water and actively interacts with the titanium and aluminum base during the vacuum sintering at 1,000 °C. In the case of a mixture with aluminum (*Al* + chips (*steel 45*)), the sintering actually is running in the thermal explosion mode, and the results of interaction can be in the form of a multiphase powder product with the synthesized Al_2O_3 oxide phase. At the selected ratio of components (*Ti* + chips (*steel 45*)), interaction with titanium does not lead to any change in the phase composition, although there is a potential to increase the fraction of oxygen due to the greater oxidation of the chip. When using titanium and aluminum as a matrix material with the addition of the grinded chip (*Ti* + *Al* + chips (*steel 45*)), in-situ synthesis of the Al_2O_3 phase is not excluded if the ratio of components and the level of oxidation of the used steel chip are selected. In this case, there is the development prospect of a new composite material with a fine oxide phase in a metal matrix.

Preliminary results of the features analysis of the metal-matrix composites structure formation with the participation of the steel chip under vacuum sintering conditions showed that metalworking waste can be used as an interacting component of the powder mixture after appropriate technological preparation. Further studies will make it possible to determine the working range of the component concentrations, its optimal ratio, which can ensure the certain structural-phase state formation that predetermines the corresponding properties.

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Conflicts of Interest

The authors declare no conflict of interest.