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Research article
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Evolution of the structural-phase state of steel swarf during its processing into a powdered product

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Abstract. Industrial waste recycling is not only linked to significant environmental challenges but also to the recovery of material resources. Typically, these recovered materials are reused within the same technological niche where the waste was generated, often through remelting or adding them to the charge. This study presents an alternative approach that enables the production of a functional powder product from steel swarf during the recycling process, which can subsequently be utilized in the creation of powder metal matrix composites. The initial structure of the swarf, following the turning of a steel billet, was examined using scanning electron microscopy (SEM) and *X*-ray diffraction (XRD) analysis after a processing complex involving additional oxidation and grinding. This analysis aimed to assess the degree of transformation in the structural-phase state of the steel swarf during its processing. It was observed that the swarf post-turning exhibited a complex morphological structure with an uneven distribution of oxygen and carbon. The oxygen present in the initial state of the swarf was insufficient to form a noticeable volume of oxides detectable by *X*-ray diffraction. However, SEM revealed individual oxide inclusions, each no more than 5 μm in size, located sporadically. Additional oxidation followed by grinding in a vibrating mill altered the structure of the steel swarf, increasing the volume fraction of oxide phases. The study's findings indicate that the resulting powder from recycled steel swarf is essentially a metal matrix composite with oxide inclusions based on an iron matrix, which holds potential for various future powder technologies.

Keywords: steel swarf, grinding, oxidation, structure, iron oxides, composite powders, sintering

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Эволюция структурно-фазового состояния стальной стружки в процессе ее переработки в порошкообразный продукт

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Аннотация. Утилизация отходов промышленного производства связана не только с решением экологических проблем, но и с повторным использованием материальных ресурсов. Чаще всего возвращаемые в производство материальные ресурсы стараются применить в той же технологической нише, где формировались сами отходы, через их переплавку или добавление в шихту. В данной работе предлагается альтернативный подход, позволяющий при утилизации стальной стружки получать функциональный порошковый продукт, который можно в дальнейшем использовать при создании порошковых металломатричных композитов. С помощью растровой электронной микроскопии и рентгенофазового анализа была исследована структура стружки в исходном состоянии (после токарной обработки заготовки из стали 45) и после дополнительного комплекса обработки (окисления и измельчения) с целью оценки степени трансформации ее структурно-фазового состояния в процессе переработки. Показано, что стружка после токарной обработки имеет сложный морфологический вид с неоднородным распределением кислорода и углерода. Растровая электронная микроскопия исходного состояния стружки позволила выявить отдельные включения оксидов размерами не более 5 мкм в удаленных друг от друга локальных местах. Однако небольшой совокупный объем и индивидуальный размер оксидных включений затруднили идентификацию этих фаз с помощью рентгенодифракционного метода. Дополнительное окисление с последующим измельчением в вибромельнице трансформирует структуру стальной стружки, повышая объемную долю оксидных фаз. Результаты проведенных исследований показали, что полученный порошок из переработанной таким образом стальной стружки представляет собой фактически металломатричный композитный материал с оксидными включениями на основе железной матрицы, который можно использовать в дальнейшем в разных порошковых технологиях.

Ключевые слова: стальная стружка, измельчение, окисление, структура, оксиды железа, композиционные порошки, спекание

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Introduction

In the framework of resource efficiency and waste utilization strategies, powder metallurgy technologies are highly preferable. This is particularly evident in the recycling of metallic waste within the engineering sector [1–3]. Metal processing on various machines contributes significantly to the total volume of manufacturing waste. Regardless of the processing type and tools used, metal swarf is always produced when manufacturing any part and is often recycled as metal scrap in metallurgical processes [2; 3; 5–7].

Traditionally, metalworking waste is reused either in remelting processes or as a charge for bulk powder blanks and coatings [1; 2; 4–13]. The range of materials studied is broad, encompassing traditional steels, cast irons [5; 6], non-ferrous metals, and alloys [13–15]. Typically, the first stage of recycling metalworking waste involves cleaning it of impurities and contaminants [16–19]. These primarily include organic contaminants from various lubricating-cooling fluids (LCF) and excess oxygen from oxidation processes during machine processing and waste storage [16]. However, these “harmful” impurities and contaminants can be sources of additional elements for forming a functional multicomponent structure in the recycled waste, ultimately transforming it into a powder form.

Several factors motivate this task. First, the swarf structure differs from the original metal billet due to the defect structure formed during cutting [20]. Second, processing modes and environments, including coolant use and oxidation processes, significantly influence it. Third, the swarf is a sufficiently activated material that can undergo additional processing, including oxidation and crushing, to convert it into a powder form [19; 21; 22].

Considering waste from processing steel billets, the resulting swarf can serve as a convenient raw material for preparing powder compositions with a specific combination of components. For example, excess oxygen and oxidation products in the waste from processing steel billets can form oxide inclusions in the steel matrix. The proposed combination of components ($Fe + Fe_2O_3 + Fe_3O_4 + FeO$) can act as a precursor for further use in other multicomponent powder compositions [21–23]. The oxide phases formed after processing steel swarf, along with the iron itself, can actively interact with titanium, aluminum, or other elements [24; 25], creating prospects for developing new metal-matrix composites. Converting steel swarf fragments into powder with a specific phase composition, morphology, and particle dispersion for further use in powder mixtures is essential.

Various methods can grind steel swarf, including electro-physical [8] or simple mechanical methods [19]. Depending on the intended application of the powder blanks, the technological parameters of crushing are determined to obtain powder particles of the required sizes from the recycled waste. Mechanical grinding using crushers or vibratory mills with steel balls is most common [19; 21]. The swarf structure transforms at each processing stage.

Unfortunately, there are currently few studies in this area, making it challenging to predict the possible evolution of the structural-phase state of metalworking waste for further use. If the products of processing steel swarf are considered potential components in powder mixtures, it is interesting to study the evolution of the structure of steel swarf from the initial turning process to converting it into powder form using additional oxidation and grinding. Such analysis is significant in developing new composite materials with oxide inclusions, which was the aim of this study.

Materials and methods

Steel billets made from carbon steel 45, the most common alloy in engineering production, were used as the research material. Its chemical composition according to GOST 1050-2013 is as follows (wt. %):

C	0.42–0.50
Si	0.17–0.37
Cu	≤ 0.25
As	≤ 0.08
Mn	0.50–0.80
Ni	≤ 0.25
P	≤ 0.035
Cr	≤ 0.25
S	≤ 0.04

Steel swarf was obtained at a machine-building enterprise after the facing operation without using lubricating-cooling fluids (LCF). Its general appearance in the initial state, microstructure of its fragments after etching with a 4 % aqueous solution of nitric acid, phase composition, and surface morphology after machining are shown in Fig. 1. It is worth noting that the results of *X*-ray phase analysis of swarf from steel 45 did not reveal lines of oxide phases, showing a practically standard set of phases characteristic of this grade of steel (Fig. 1, *d*), consistent with previous results [26]. The size of swarf fragments after machining the steel billet was 3–7 mm in width and 10–30 mm in length, necessitating grinding using a vibratory mill designed by ISPMS SB RAS (Russia). Grinding was carried out in an air environment for 10 hours with steel balls of 15 mm diameter at a swarf-to-balls mass ratio of 1:30.

Despite the machining conditions and associated processes of temperature fluctuations, oxygen saturation, and work hardening, the swarf remained ductile, complicating the grinding process. Therefore, additional oxidation of the swarf fragments was employed, increasing their brittleness and enabling the production of powder with a wide range of particle dispersity – from 50 to 350 μm. To assess the compressibility and sinterability of the resulting powder, cylindrical samples 10–13 mm high and 10 mm in diameter were pressed and sintered in a vacuum furnace at temperatures ranging

$$\theta = \left(1 - \frac{\rho_{smp}}{\rho_{theor}}\right) \cdot 100\ \%,$$

where ρ_{smp} is the actual density of the sample (g/cm^3), and ρ_{theor} is the theoretical density calculated by the additive method for powder particles from the recycled steel swarf, assuming at least 30 vol. % oxide phase and the remainder as a solid solution based on iron corresponding to steel 45.

Since the quantitative values of the oxide fraction are averaged, determining the exact theoretical density is challenging. Therefore, to assess the pore content, the quantitative metallography method was additionally used. The structural-phase changes in the steel swarf were investigated by comparing the results of structural and elemental analyses and the morphology of its fragments in the initial state after turning the steel billet and a set of subsequent actions – oxidation, grinding, pressing, and vacuum sintering.

The studies were conducted using optical metallography, *X*-ray phase analysis (XRD), and scanning electron microscopy (SEM) with energy-dispersive

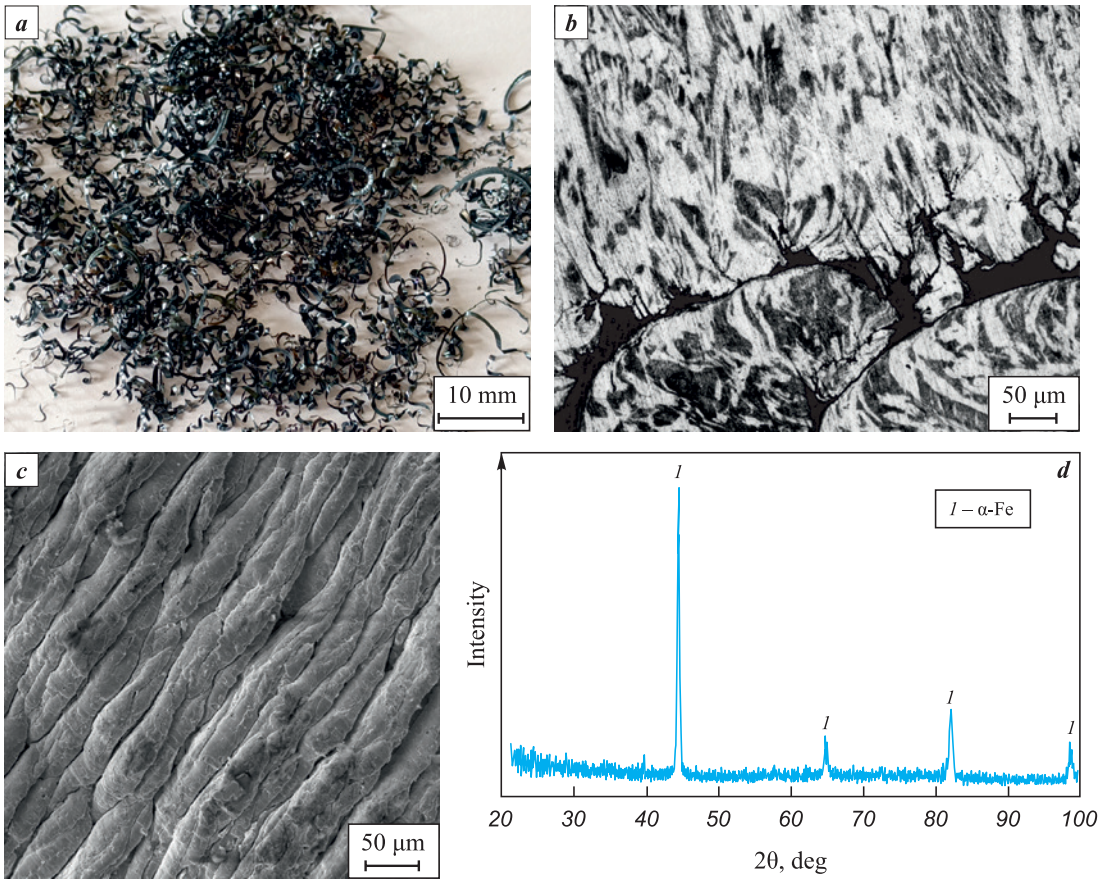

Fig. 1. General appearance of steel swarf (***a***), microstructure of its fragments (***b***), SEM image of one of the surface areas (***c***), and their phase composition (***d***) after machining

Рис. 1. Общий вид стружки из стали 45 (***a***), микроструктура ее фрагментов (***b***), РЭМ-изображение одного из участков поверхности (***c***) и фазовый состав после металлообработки (***d***)

microanalysis. These analyses were performed using the equipment of the Tomsk Regional Shared-Use Center of NR TSU and the Shared-Use Center of ISPMS SB RAS. The specific equipment used included: optical microscope AXIOVERT-200MAT (Carl Zeiss, Germany), *X*-ray diffractometers XRD (Shimadzu 6000, Japan) and DRON-8 (NPP Burevestnik, Russia), and scanning electron microscope MIRA 3LMU (TESCAN, Japan).

Results and discussion

The preliminary analysis of the steel swarf showed that after machining without using lubricating-cooling fluids (LCF), a highly defective structure formed on its surface (Fig. 2). Elemental analysis revealed an uneven distribution of carbon and oxygen (Fig. 2, *c*, *d*). The noticeable presence of oxygen on the swarf surface suggests the formation of a certain volume of iron oxides. However, the XRD analysis did not detect oxide phases (Fig. 1, *d*) in sufficient quantity for identification.

It can be assumed that the oxidation processes during initial machining primarily contribute to the formation of amorphous oxide films, which are not visible to XRD, preventing the formation of iron oxide crystallites in significant quantities. Scanning electron microscopy at high magnification revealed individual inclusions that can be attributed to iron oxides based on the elemental distribution map in the local area of the swarf surface (Fig. 3). However, their size and quantity fall below the sensitivity threshold of the *X*-ray diffractometer.

Since the steel swarf remained ductile after turning, and the oxide phase content was minimal, it was decided to further oxidize the swarf fragments. This aimed not only to increase the iron oxide content but also to enhance the swarf's brittleness. Thus, the oxidation process would achieve two goals: effective swarf grinding and increased iron oxide content. The simplest methods for this were used: heating in air or soaking in water followed by drying.

Despite the straightforward task of oxidizing carbon steel swarf, the specifics of iron oxidation must be

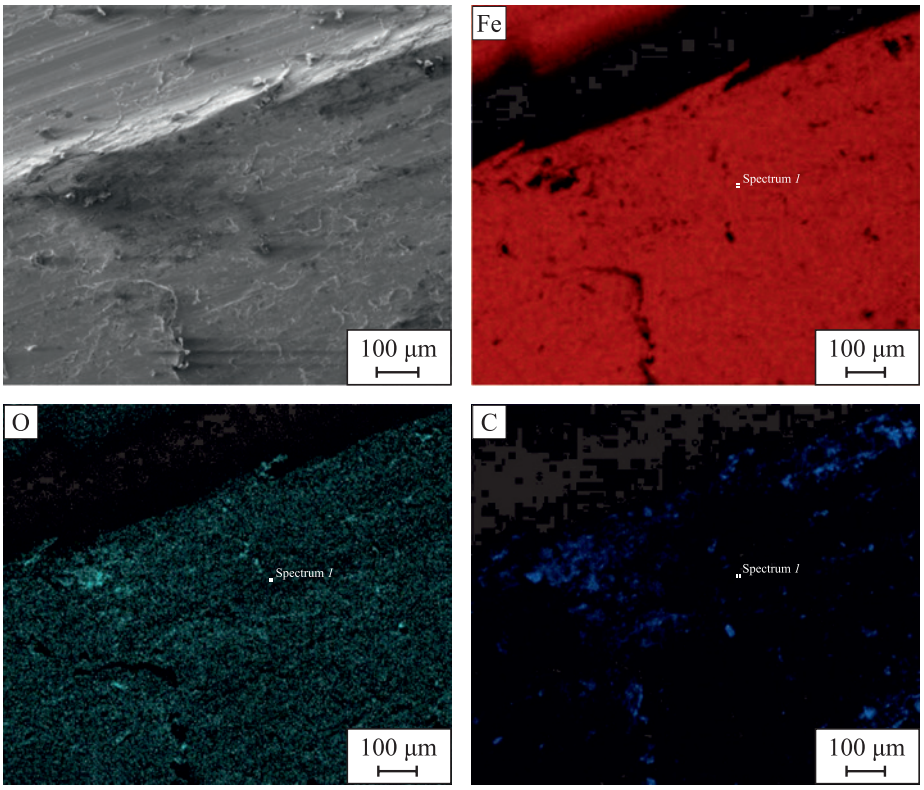

Fig. 2. SEM image of the cut surface and elemental distribution maps of iron, oxygen, and carbon in the steel swarf after processing the steel 45 billet

Рис. 2. РЭМ-изображение поверхности среза и карты распределения железа, кислорода и углерода в стальной стружке после обработки заготовки из стали 45

considered. Iron forms several oxides: FeO, Fe_2O_3 and Fe_3O_4. The oxidation process also goes through several stages, where one oxide can transform into another under certain conditions or act as a barrier to its formation. There are also specific temperature regimes that predetermine the formation of a particular group of oxides [27].

When heating steel swarf in a muffle furnace in air up to 400 °C, XRD results showed that such thermal treatment not only removed the work hardening from

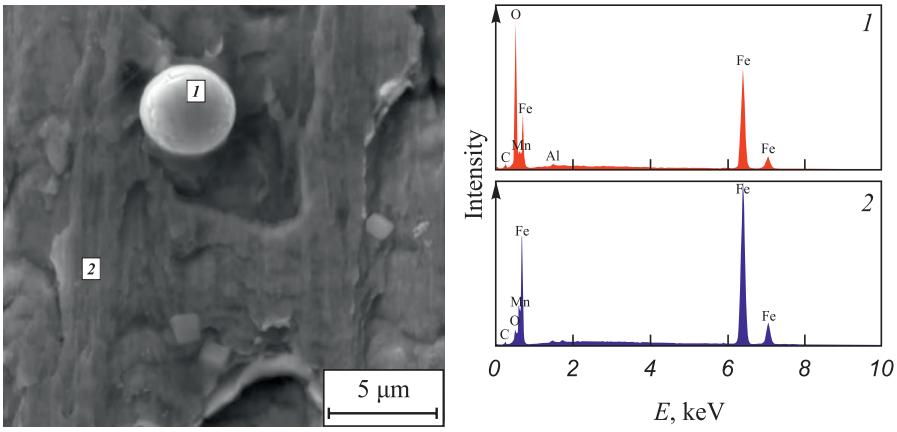

Fig. 3. SEM image (×10,000) and EDX spectral analysis results of local points on the surface of the steel swarf

1 – area corresponding to the iron oxide spectrum; ***2*** – area of the base material

Рис. 3. РЭМ-изображение (×10 000) и результаты спектрального анализа (ЭДС) локальных точек на поверхности стальной стружки

1 – область, соответствующая спектру оксида железа; ***2*** – область основного материала

machining, which hindered grinding in the vibratory mill but also formed carbide-containing phases based on Fe_2C, FeC, $FeCO_3$ or $Fe(CO)_5$ (Fig. 4, *a*). Since the lines of these phases were few and their intensity very weak, correctly identifying these phases is challenging and requires separate investigation. It is assumed that a mixture of carbide or oxycarbide phases primarily formed on the surface of the swarf fragments may be present.

The main goal of the experiment was to test different methods to increase the iron oxide content in the steel swarf. Therefore, *X*-ray phase analysis focused on identifying those oxides not detected by XRD after thermal treatment.

Another oxidation method involved soaking the swarf in water for at least 48 h. Wetting with water followed by air drying at room temperature allowed XRD to detect up to 30–40 vol. % iron oxides within 48 h (Fig. 4, *c*). There remains a challenge in distinguishing the phases of magnetite Fe_3O_4 and γ-Fe_2O_3 by *X*-ray diffraction, requiring further study. Additionally, an option was explored to obtain powdered fragments from swarf waste by precipitating the suspension of ground steel 45 swarf products from water after evaporation (Fig. 4, *b*).

After drying the aqueous solution, a fine powder was obtained. Phase analysis revealed a complex configuration of iron dihydroxycarbonate $Fe_2(OH)_2CO_3$ in the presence of iron oxides. Since this powder product had a very complex composition (requiring separate study) and the productivity of this method was significantly lower than that of the conventional method of soaking in water, drying, and subsequent grinding, further research focused on the sifted powder from the ground swarf after soaking in water.

Structural-phase and elemental analyses of the preoxidized and ground swarf showed that during

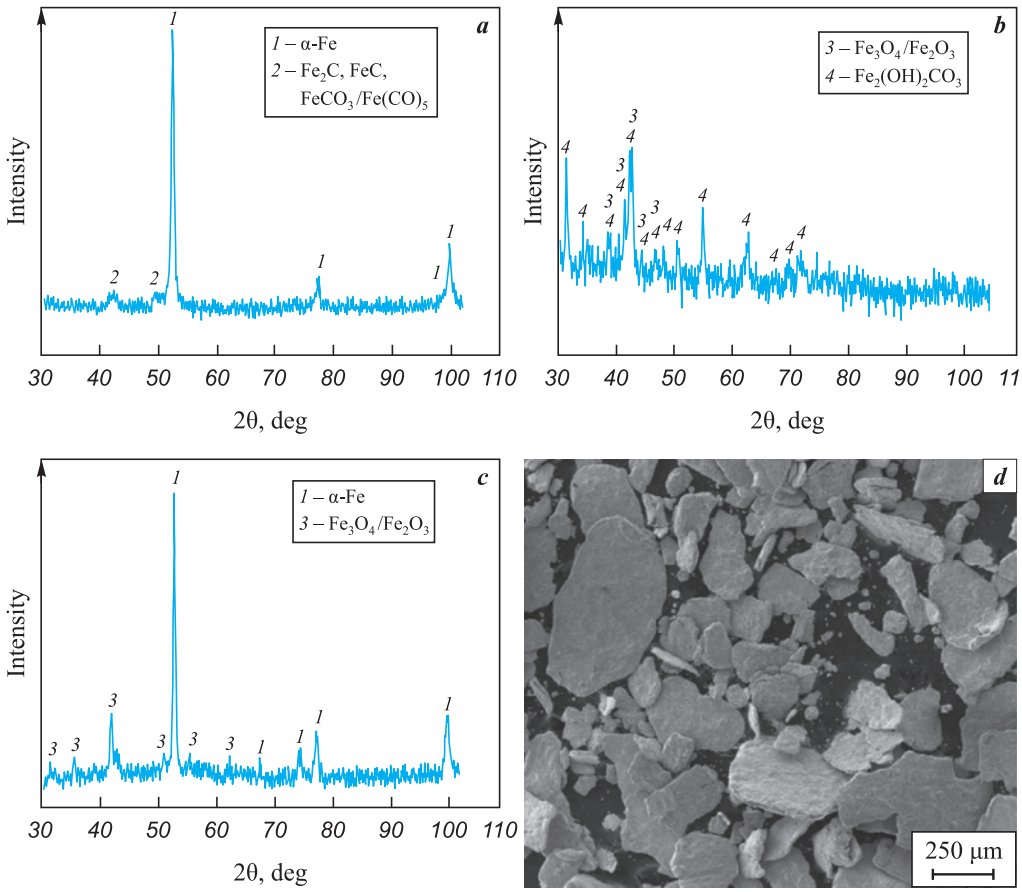

Fig. 4. Phase composition (***a***–***c***) and general appearance (***d***) of metal ground steel 45 swarf

a – swarf after annealing in air in a muffle furnace at 400 °C;
b – precipitated suspension after evaporation from an aqueous solution with steel swarf;
c and ***d*** – swarf after soaking in water for 48 h and drying at room temperature

Рис. 4. Фазовый состав (***a***–***c***) и общий вид (***d***) измельченной металлической стружки из стали 45

a – стружки после отжига на воздухе в муфельной печи при t = 400 °C;
b – осажденной взвеси после испарения из водного раствора со стальной стружкой;
c и ***d*** – стружки после выдержки в воде в течение 48 ч и сушки при комнатной температуре

the selected comprehensive treatment with oxidation in water and intensive mechanical crushing, the fragments of steel 45 swarf transform into a composite metal matrix powder material (Fig. 5). The particles consist of an α-Fe matrix, with iron oxides Fe_2O_3 or Fe_3O_4 as inclusions. It is noteworthy that the oxide phases primarily form on the surface of the ground oxidized swarf fragments (Fig. 5, *a*), while the original structure is preserved inside the particles.

The swarf fragmented into particles of a wide size range – from large fragments (300–350 μm) to small ones (20–50 μm). The presence of a significant amount of oxides (at least 30 vol. %) could pose challenges for compacting the obtained powder. Therefore, samples were pressed at a low pressure (350–400 MPa) without adding any plasticizers to test this.

Despite the presence of oxides, the powder was well-compacted, and under the selected pressing load, the residual porosity of the samples was about 40 %. Subsequent vacuum sintering of the pressed samples led to a reduction in porosity due to shrinkage, the intensity of which increased with the sintering temperature (see the Table).

Relative change in volume and porosity of compacts from recycled steel swarf after vacuum sintering

Относительное изменение объема и пористости прессовок из переработанной стальной стружки после вакуумного спекания

Sintering temperature, °C	$\Delta V/V_0$, %	$\Delta\theta/\theta_0$, %
1000	5.9	4.2
1200	25.0	10.5

Despite the presence of oxides, the powder was well-compacted, and under the selected pressing load, the residual porosity of the samples was about 40 %. Subsequent vacuum sintering of the pressed samples led to a reduction in porosity due to shrinkage, the intensity of which increased with the sintering temperature (see the Table). The structural-phase state of the powder compact sintered at 1000 °C is shown in Fig. 6. XRD results of the obtained compacts indicated that the formed group of iron oxides Fe_3O_4/Fe_2O_3 transitions to monoxide FeO, with α-Fe being the primary phase (Fig. 6, *a*). The interparticle contact zones

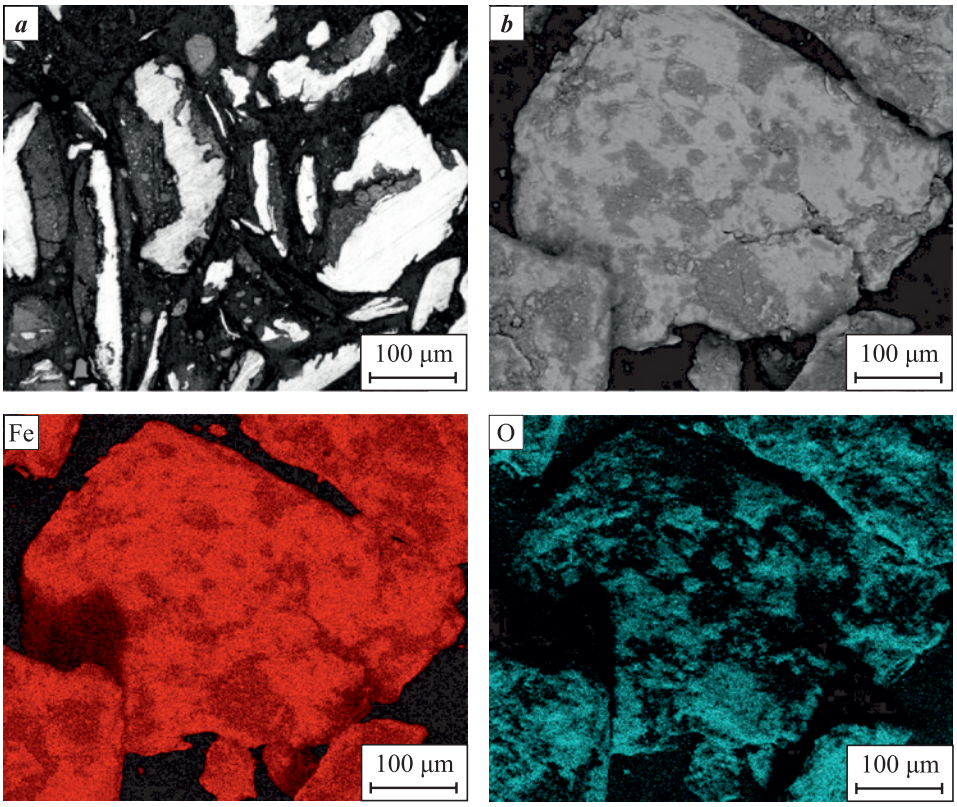

Fig. 5. Metallographic (***a***) and SEM (***b***) image of crushed particles of treated steel swarf with elemental distribution maps of iron and oxygen

Рис. 5. Металлографическое (***a***) и РЭМ (***b***) изображения дробленых частиц обработанной стальной стружки с картами распределения железа и кислорода

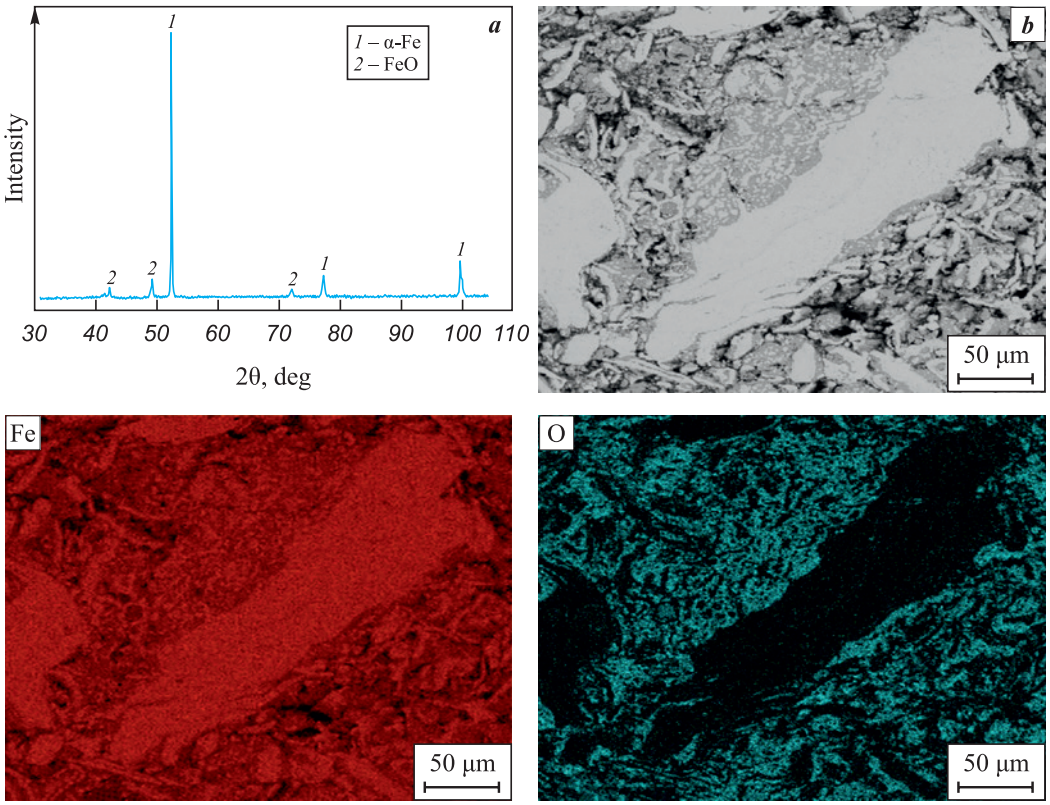

Fig. 6. Phase composition (***a***), microstructure (***b***) and elemental distribution maps of iron and oxygen in the compact sintered at 1000 °C from recycled steel swarf powder

Рис. 6. Фазовый состав (***a***), микроструктура (***b***) и карты распределения железа и кислорода в спеченной при t = 1000 °C прессовке из порошка переработанной стальной стружки

are predominantly filled with oxide components, while the main particle area is almost free of oxygen (Fig. 6). This structural configuration hinders the sintering of fragmented steel swarf particles due to the oxide-containing periphery acting as a barrier. The oxygen-rich extended regions in the recycled swarf powder particles can potentially play an active role in contact interactions with other powder components, such as aluminum or titanium [24], and participate in accompanying reduction reactions or intermetallic synthesis, which is a separate research topic. It is worth noting that the structural-phase state formed in the recycled steel swarf powder could be of interest for other prospective uses [28; 29], especially where the presence of iron oxides is relevant.

Conclusions

1. Steel swarf after turning operations on steel 45 billets exhibits specific structural features due to the deformation and physicochemical processes associated with machining. In its initial state, it demonstrates a structure with an uneven distribution of carbon and oxygen, localized in certain areas as fine inclusions within the steel matrix.

2. The XRD analysis of the swarf in its initial state revealed a phase composition identical to that of the steel 45 billet, with the swarf fragments remaining ductile. Additional oxidation of the metalworking waste in water promotes the growth of the oxide phase and facilitates the grinding process of the swarf in a vibratory mill, yielding particles sized 50–350 μm.

3. The analysis of the resulting powders from the oxidized and ground steel 45 swarf in the vibratory mill showed that the powder particles are a metal matrix product with oxide inclusions predominantly in the surface layers. Despite the presence of at least 30 vol. % iron oxides, the powder is well-pressed and sintered, demonstrating volumetric shrinkage and reduced porosity.

4. The elongated regions of oxide phases formed in the ground swarf fragments are active structural inclusions that can interact with additional powder components containing other elements, such as aluminum or titanium. This allows the investigated powder from steel swarf to be considered a potential precursor or oxide-containing component for producing multicomponent metal matrix composites with an oxide phase, utilizing waste from the machine-building industry.

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I. O. Nikolaev – prepared the experimental material and samples, conducted structural studies, participated in the discussion of the results, and prepared the illustrations.

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