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THE TECHNOLOGY OF OBTAINING POWDERS USED IN THE PRODUCTION OF SOLID ALLOYS

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Abstract. The scientific and practical study on contemporary technologies and methods for generating powders (cobalt and tungsten carbide)-the building blocks of tungsten carbide-cobalt-based solid alloys that are widely utilized in the mining and metallurgy industries-is presented in this article. In the course of research, it was studied how to obtain pure cobalt powder from cobalt oxide and pure tungsten carbide powder from tungsten oxide. Cobalt is a binder that affects the hardness, brittleness, density, bending strength, tempering temperature and abrasive wear resistance of hard alloys. The binder is required to be sufficiently flexurally strong, tough and resistant to abrasive wear under operational conditions and this requires obtaining pure cobalt powder. Today, the main raw materials for the production of hard alloys at the "SPA to produce rare metals and the hard alloys" under "Almalyk MMC" JSC are brought from the waste cakes in the territory of the SPA and the Ingichka mine located in the Samarkand region and hydrometallurgically processed tungsten oxide takes up to (WO_3).To solve this task, the electron microscopic method was used during the research. All studies were performed on a JSM-IT200 (JEOL, Japan) scanning electron microscope at the "Uzbekistan-Japan Youth Innovation Center".

Keywords: *Cobalt powder, cobalt oxide, tungsten carbide, solid alloys.*

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1. Introduction

More importantly, the technique of powder acquisition has a key importance in fabrication of solid alloy that is useful industrially because of the mechanical and chemical characteristics. Casting and forging are traditional conventional techniques of metal forming, while lineage from metal powder to materials and constituted components is known as powder metallurgy which has many benefits as compared to casting and

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forging processes (Yang *et al*., 2024; Capus, 2000). Besides enabling the development of complex shapes and geometries with least wastage of the material, this method also offers a direct manipulation of degree of alloyage (Solberg, 2016).

Thus, in recent decades, improvements in technologies for producing powders have greatly improved the performance and versatility of solid alloys. Conventional production methods including atomization, mechanical alloying and chemical reduction have been optimized for size distribution, powder purity and morphology that determines the properties of the alloy. These powders are the base for synthesizing high-performance solid solutions needed in industries such as aerospace, automobile, biomedical and power (Yuan *et al*., 2019).

The nature of the powder affects the microstructure and mechanical characteristics of the alloy. Peculiarities in the preparation of the powder which include particle size and size distribution, geometric shape and purity have significant influence on the sintering process and the performance of the alloy (Wendt, 2021). Therefore, current research work is devoted to fine-tuning these properties of metal powder so as to tailor the desirable characteristics of the alloy that it will form, such as strength, hardness, wear and corrosion resistant (Katz-Demyanetz *et al*., 2019).

The carbidization process is produced by reacting tungsten powders with carbon at high temperatures in a hydrogen atmosphere (Reddy *et al*., 2021). The chemical reactions shown below were occurred during the process of carbidization:

$$W+C=WC$$
$$C+O_2=CO_2$$
$$CO_2 + C=2CO$$
$$W+2CO=WC+CO_2 .$$

The tungsten carbide production process consists of the following operations: preparation of raw materials for the carbidization process; carbidization process and sifting (Chkhartishvili *et al*., 2023; Parmonov *et al*., 2024). Preparation of the mechanical mixture for the carbidization process, which consists of technical brand T-900 carbon powder, tungsten powder and if necessary, cobalt powder, which acts as a binder, is one of the most important processes in the technology of obtaining tungsten carbide-cobalt-based solid alloys by the powder metallurgy method (Bose, 2003). Therefore, the research goal of this process was to obtain well-mixed tungsten carbide mixtures with essentially uniform size distribution of the constituents.

2. Material and Methods

In the process of sintering tungsten carbide-based hard alloys, one of the factors affecting the hardness, brittleness, density, bending strength, sintering temperature and abrasive resistance available in the hard alloys is the binder properties (Zhang *et al*., 2018). The binder is required to be sufficiently flexurally strong, tough and resistant to abrasive wear under operational conditions. At the same time, during annealing, the binder should soak well and partially dissolve the carbide phase (Meshalkin *et al*., 2020; Farmonov *et al*., 2024). The above requirements are fully satisfied by iron group metals Co, Ni and Fe. Today, nickel and iron powders are used as binders in powder metallurgy instead of cobalt, whose price is relatively banal (Abbott, 2004).

The fact that nickel is more common than cobalt, the formation temperature of the system eutectic is at a relatively low temperature and it is more resistant to oxidation than iron and cobalt at high temperatures is the reason why it has been widely used as a binder in the production of tungsten and tungsten-free hard alloys in recent years (Lassner, 2012; Parmonov *et al*., 2024).

The reason why nickel reacts with tungsten carbide during annealing and the degree of densification proceeds much faster than in the tungsten carbide-cobalt system is due to the formation of eutectic nickel at relatively lower temperatures. In Tungsten Carbide Cobalt and Tungsten Carbide Nickel based hard alloys, carbide particle growth starts at 1400℃, but the optimal annealing procedure for the Tungsten Carbide Nickel system is a much wider range than Tungsten Carbide Cobalt system (French, 1983).

As a result of the insufficient dissolution of tungsten carbide particles in nickel binder, titanium carbide-nickel-based hard alloys exhibit lower strength and abrasive wear resistance than tungsten carbide-cobalt based hard alloys and conglomerates of carbide particles are formed. The resulting conglomerates additionally increase the brittleness of the hard alloy (Berger, 2015). At the same time as the brittleness decreases, the hardness of the surface layer of hard alloy particles decreases slightly due to the defective transformation of carbon in the outer layers of tungsten carbide particles (Testa *et al*., 2020).

The conducted literature analysis and scientific-practical experiments showed better mechanical properties of samples obtained using cobalt as a binder than nickel and iron. For this reason, we used cobalt powders in all our experiments.

3. Results and Discussion

Research on the technology of obtaining cobalt powder from cobalt oxide. Cobalt powders, which are utilized in the production of hard alloys with corrosion-resistant, are imported products in the form of cobalt (III) oxide to the “SPA for the production of rare metals and hard alloys” under “Almalyk MMC” JSC. Extraction of cobalt powder from cobalt (III) oxide was carried out in a hydrogen atmosphere in two stages in STN-2.35 electric furnaces. The recovery process takes place in the following reaction:

$$Co_2O_3 + 3H_2 = 2Co + 3H_2O\ .$$

Initially, cobalt oxide is loaded into boats until they are full and boats filled with cobalt oxide powder are loaded into the electric furnace one after the other. The speed of movement of boats in the oven is 12-20 mm/min. 6-12 m^3 of hydrogen per hour is supplied along the length of the furnace from the back and the boats move towards the hydrogen flow with the help of the furnace's mechanical thruster. The furnace consists of 2 zones: in zone 1, the temperature is 500-600℃ and in zone 2, the temperature is 600-750℃ (initially, the temperature in the furnace zones, the speed of movement of boats in the furnace and the amount of hydrogen are determined depending on the density of the cobalt oxide powder) (Luidold & Antrekowitsch, 2007). The powder passed through 2 stages is taken out of the oven after it has completely cooled down in the cooling stage. Taking powders out of the oven before they have cooled down completely will cause the cobalt to oxidize again:

$$4Co + 3O_2 = 2Co_2O_3$$
$$2Co + O_2 = 2CoO\ .$$

After complete cooling, the cobalt powder removed from the furnace is ground in a ball mill at a speed of 30-40 rpm for 30 to 120 minutes, depending on the particle size, to ensure granularity (Dayton, 1982).

Table 1. The chemical structure of the PK-1u brand of cobalt

Co, at least, %	Amount of additives in the composition, % max.					
	Fe	Si	Ni	C	Cu	Loss of H_2 during calcination
99.25	0.2	0.025	0.4	0.02	0.04	0.1

Research on the technology of restoring tungsten oxide to the tungsten state. Today, the primary raw materials utilized by the PB to produce hard alloys and rare metals under "Almalyk MMC" JSC are brought from the waste cakes in the territory of the SPA and the Ingichka mine located in the Samarkand region and hydrometallurgically processed tungsten oxide takes up to (WO_3). The chemical composition of hydrometallurgically processed tungsten oxide is presented in Table 2.

Table 2. The chemical structure of WO_3

	Naming	Detectable elements, %										
		Fe	Al	Si	Ca	Mg	Mo	Ni	As	Spish	P	S
1	W_2O_3	0.013	0.008	0.011	0.058	0.28	0.11	0.002	0.003	0.33	0.002	0.002
2	W_2O_3	0.024	0.002	0.01	0.057	0.34	0.11	0.002	0.003	0.37	0.002	0.003
3	W_2O_3	0.023	0.012	0.01	0.020	1.7	0.28	0.007	0.003	0.37	0.002	0.003

The chemical composition of the tungsten oxide that has undergone the restoration process after restoration to the tungsten state is presented in Table 3.

Table 3. Chemical composition of the tungsten oxide

	Naming	Detectable elements, %										
		Fe	Al	Si	Ca	Mg	Mo	Ni	As	Spish	O_2	S
1	W-powder	0.028	0.001	0.019	0.047	0.45	0.2	0.005	0.003	4.4	0.09	0.003
2	W-powder	0.04	0.002	0.021	0.033	0.7	0.14	0.005	0.003	4.9	0.12	0.003
3	W-powder	0.017	0.002	0.018	0.035	0.4	0.24	0.015	0.003	8.0	0.33	0.003

It can be seen that the magnesium content is higher in WO_3 powders and W powders (Tables 2 and 3). The research revealed that magnesium in the raw material is present in the structure that has undergone the annealing process as the final process. This causes a sharp drop in all the properties of hard alloys. Magnesium in the raw material should be removed during the carbitization process.

a b c

Figure 1. WO_3 powder loaded into the boat (a), process of loading the boat into the furnace (b) and W powder coming out of the furnace (c)

Preparation of raw materials for the carburizing process. For the production of various types of carbides, the components of the aggregate are mixed for 2 hours according to Table 4 by weight. The powder components were mixed in the following order (for 1 kg of the powder mixture): according to the selected composition (W - powder and C - powder), each powder component was weighed on a BA-W1203 electronic scale with an accuracy of 0.001 g; the powder components were mixed with each other in a ball mill for 2 hours, to determine whether the powders were completely mixed with each other, chemical analysis was carried out using a JSM-IT200 brand scanning electron microscope.

During the mixing process, It was confirmed that the mixture's total carbon content fell between 6.00 and 6.07% by mass. Tables 4 and 5 show the chemical makeup of the tungsten powders that were chosen as raw materials.

Carbidization of tungsten powders is carried out in hydrogen flow in electric resistance furnaces with graphite tubes (Figure 2) or in 2-zone muffle furnaces with molybdenum heaters. Due to the good quality of tungsten carbide powder obtained from electric resistance furnaces and the absence of foreign additives, the carbidization process was carried out in an electric resistance furnace with graphite tubes.

First, raw materials are loaded into boats, boats are supplied from the front of the furnace and hydrogen is supplied from the back of the furnace. Boats move at a speed of 25-30 mm per min. The carbidization process was carried out according to the regimes shown in Table 6.

Table 4. Mixing the components of the slag by weight to produce different types of carbides

Device	Product		Loaded balloon		Mixing time, hours	The rotational speed of the ball mill, rev/min	Hard alloy brand
	W, kg	C %	Ø, mm within	Weight, kg			
Ball mill (V-380 l)	200	6.0	15-35	250-300	2	33-40	For the VK group
	200	6.04	15-35	250-300	2	33-40	For the VK group
	200	6.07	15-35	250-300	2	33-40	For the VK group

Table 5. The chemical composition of W obtained for the carbidization process

	Naming	Detectable elements, %										
		Fe	Al	Si	Ca	Mg	Mo	Ni	As	O_2	S	Particle size, mm
1	W-powder	0.028	0.001	0.019	0.047	0.45	0.2	0.005	0.003	0.09	0.003	4.4
2	W-powder	0.04	0.002	0.021	0.033	0.7	0.14	0.005	0.003	0.12	0.003	4.9
3	W-powder	0.017	0.002	0.018	0.035	0.4	0.24	0.015	0.003	0.33	0.003	8.0

Table 6. Carbidization process procedure in graphite tube electric resistance furnace

Temperature, °C	**Boat speed, mm/min**	**Hydrogen consumption, m^3/h**	**Naming**
1800±50	25-30	0.5-1.5	For hard alloy VK4
2000±50	25-30	0.5-1.5	For VK-VK hard alloy
1800±50	8-10	0.3-0.5	For VK-V hard alloy

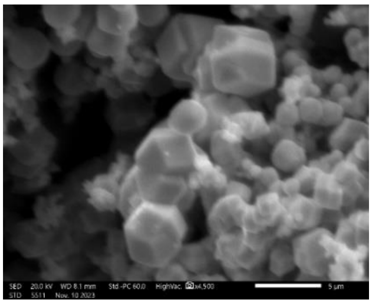
According to the results of the research, the samples with low carbon content were added to the solid materials depending on the total carbon content and the repeated mixing process was carried out. In most studies, this process has led to negative results and such compounds have produced carbides with varying amounts of total carbon. Some samples yielded carbides with a total carbon content greater than 6.12% by weight and in other samples less than 6.08%. Naturally, tungsten carbide-cobalt-based hard alloy products have different amounts of total carbon and hard alloy samples with different properties were obtained from them (Vahlas *et al.*, 2006).

If the carburizing process and mixing process of the components of the mixture are done correctly, there will be no shortage or excess of carbon. This can only be due to the mixing factor, that is, the components of the mixture are poorly mixed. In one place, carbon may not be in the required amount and in another, it may accumulate excessively. A scanning electron microscope study of the raw material mixture revealed the availability of areas with a small amount of carbon powder particles. The presence of areas with carbon powder particles was caused by poor mixing of carbon powder and tungsten powder before the carbidization process. Therefore, one should not make hasty conclusions about the quality of the finished product based on the only analysis performed during the carbide production process (chemical analysis to determine the total carbon content and composition) and any components to bring the carbon content to the desired level of the semi-finished product and even more (tungsten or carbon) will need to be added. To do this, it is necessary to repeat the raw material mixing procedure and then repeat the chemical analysis of the total carbon in the mixture (Goeppert *et al.*, 2014).

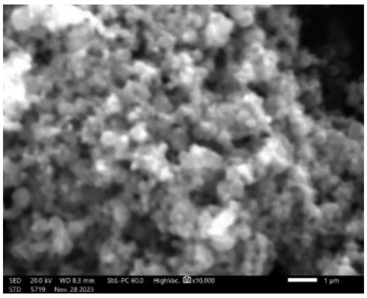
During the conducted studies, it was considered that the total carbon content of the samples was 6.00-6.0.7% and they were differentiated by the range of Δ. If Δ is small, the components of the mixture are evenly distributed throughout the volume. Therefore, if $\Delta \leq 0.05\%$ and Cum.=6.00-6.07%, the mixture is considered to be of good quality and the carbidization process is carried out. If $\Delta > 0.05\%$ and Cum. = 6.00-6.07%, the mixture is repeatedly mixed for 1 hour and the process is carried out until the value of $\Delta \leq 0.05\%$ is reached. The chemical structure of the tungsten carbide powders is presented in Table 7.

Table 7. The chemical composition of WC obtained after the carbidization process

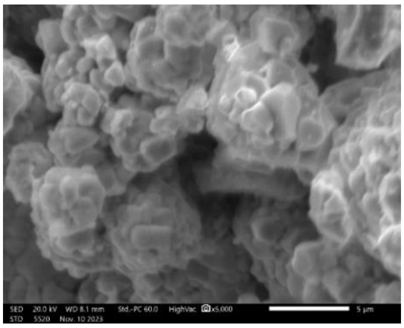
	Naming	Elements being identified, %					
		W	C_{common}	C_{free}	Fe	Mg	Other
1	WC-powder	93.860	6.00	0.024	0.10	0.015	0.001
2	WC-powder	93.805	6.00	0.02	0.17	0.017	0.003
3	WC-powder	93.577	6.20	0.015	0.17	0.014	0.002
4	WC-powder	93.646	6.20	0.02	0.12	0.013	0.001
5	WC-powder	93.577	6.20	0.017	0.19	0.014	0.002
6	WC-powder	93.189	6.30	0.043	0.45	0.015	0.003



a) tungsten powder (x 4 500)



b) carbon powder (x 10 000)



c) tungsten carbide powder (x 5 000)

Figure 2. Surface morphology of powders used for the carbitization process

To get rid of magnesium in the raw material, the temperature of the carburizing process was increased to 1800°C. As a result, the amount of magnesium decreased to 0.015% on average.

The morphology of tungsten, carbon powders and obtained tungsten carbide powders was studied in the process of carbitization (Otakuziyev *et al*., 2023; Dissanayake *et al*., 2023; Xiao *et al*., 2022, Abdullayev *et al*., 2024). The surface morphology of the powders is presented in Figure 2.

4. Conclusion

Based on scientific and practical investigations, it was shown that powders had a total carbon content between 6.00 and 6.07%, which is the most optimal value. If the total carbon content is lower than 6.00 percent or higher than 6.07 percent, it was found that tungsten carbide-cobalt base hard alloy samples cause structural defects. Therefore, if $\Delta \leq$

0.05% and Cum.=6.00-6.07%, the mixture is considered to be of good quality and the carbidization process is considered to be well done.

As the technology of powder production has been developed, the producers are capable to create more homogenous alloy powders, better in sintering as well as better final properties. The ability to select different powders for nearly any type of usage has created solid alloys with greatly enhanced strength, hardness, wear and corrosion characteristics. For example, finer powders with better homogeneity have been produced due to improvement in atomization process, whereas; mechanical alloying helped in developing composition of alloys with superior properties

It was discovered that the tungsten powder utilized in the study had an average of 0.4-0.7% magnesium metal, which resulted in structural flaws in the hard alloy samples based on tungsten carbide and cobalt. By increasing the temperature of the carburizing process to 1800℃, the magnesium contained in the tungsten powder was released. As a result, the mechanical strength of the hard alloy improved. The future trends in the area powder metallurgy and alloy production are expected to be marked by increased efficiency, high precision and environmental responsibility. This research is significant in that it highlights the relations among various powder production techniques and alloy performance and creates the directions for the further development of the subject.

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