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The Influence of Different Processing Parameters on the Properties of Molybdenum Powder

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ABSTRACT: This paper presents the results of a study on the influence of different processing parameters on the properties of molybdenum powders and rods. Our work reveals the significant impact of the hydrogen humidity and temperature recovery on the bulk density and oxygen content of molybdenum powder. This approach was developed to produce metallic molybdenum powder with physical and technical parameters that are suitable for the production of wire.

KEYWORDS: molybdenum powder, rod, hydrogen, temperature recovery, oxygen, bulk density.

I. INTRODUCTION

The use of molybdenum and its alloys is widespread in various industries. However, consumers are requiring an improvement in the quality of molybdenum products even though it generally meets international standards. However, some properties, such as the chemical purity, the number of controlled impurities, the concentration, and physical and mechanical characteristics, do not always satisfy the demands of consumers.

In this study, the influence of various factors on the technical characteristics of molybdenum powder is addressed. The improvement in the chemical purity, physical and technical properties of molybdenum powder is an urgent task.

II. THE PURPOSE OF THE STUDY

The aim of this work is to develop an effective technology for the production of molybdenum powder with a minimum content of oxygen and improved physical and technical properties.

III. OBJECTS AND METHODS OF RESEARCH

The object of the study is Mch grade molybdenum powder that is used for the production of molybdenum wire and meets the requirements of the normative documents in the CIS. The chemical composition and physical properties of the molybdenum powder were determined in accordance with the methods, devices and installations in the GOST and TC requirements that appear in the CIS.

IV. RESULTS OF THE INVESTIGATIONS AND DISCUSSION

To solve this problem, we researched the production of molybdenum powder with a given dispersion and low oxygen content. One of the factors influencing the embrittlement of molybdenum prepared by powder metallurgy is the presence of interstitial impurities, the content of which can be very high (up to 0.04% O₂ and 0.003-0.001% C) [1].

Interstitial impurities form a very limited range of solid solutions with molybdenum, and exceeding the solubility limit leads to the formation of nonmetallic compounds (oxides, nitrides, and carbides) that reside at the grain boundaries. During thermo mechanical deformation, these compounds cause embrittlement of the metal. [2, 3].



Therefore, the improvement of the existing processes and development of new ones at different stages of the production of molybdenum metal powder is of primary importance. The goals include reducing the content of oxygen and other impurities and improving physical and technical properties of molybdenum metal powder.

We carried out preliminary studies to identify the causes of molybdenum metal powder contamination with impurities during the recovery and sifting processes.

It was found that the oxygen content of the molybdenum metal powder was high (in the range of 0.3% and above). To reduce the oxygen content in the molybdenum metal powder and improve its physical and technical properties, we investigated the influence of various factors.

We also investigated the effect of the humidity and temperature hydrogen recovery on the bulk density and oxygen content in the molybdenum powder.

The effect of humidity on the bulk density of the hydrogen and oxygen contents in the molybdenum powder was investigated as follows.

Molybdenum dioxide was reduced to metal powder in a muffle furnace or multitubular DB-111A and STN 1.6 furnace using regenerated hydrogen. However, during the production of the molybdenum powder, oxygen was detected. Therefore, it became necessary to check the quality of the hydrogen entering the recovery furnace after regeneration.

The recovery furnace that was used for the production of the molybdenum metal powder was completed with the installation of hydrogen regeneration capability. The recovery furnace "froze" the water vapour in the processed hydrogen. Freon was used as the cooling reagent. However, setting produces the best drying of hydrogen with a dew point of $-30\text{ }^{\circ}\text{C}$, which does not produce reaction conditions for optimal recovery. Therefore, there was a need for additional drying of the hydrogen after freezing the water vapour and bringing the dew point to $-40\text{ }^{\circ}\text{C}$.

We used synthetic zeolite, natural zeolite and alumina as adsorbents for further dehydration.

As a result of tests carried out under industrial conditions, we found that the best adsorbent for water vapour was from the spent hydrogen in artificial zeolite. The artificial zeolite worked as an adsorber 4 days after its regeneration (water vapour was removed from the zeolite by passing hot air), and it was then cooled to $-40\text{ }^{\circ}\text{C}$.

The hydrogen humidity was determined three times per shift. Samples were taken periodically from the powder after the humidity screening and averaged to determine their bulk density and oxygen content. The obtained data are summarized in the Table below.

Table

The oxygen content and the bulk density of molybdenum powder as a function of the hydrogen humidity

	Dew point, $^{\circ}\text{C}$								
	-50	-45	-40	-35	-30	-25	-22	-16	-5
Oxygen content, %	0,03	0,05	0,05	0,06	0,12	0,12	0,14	0,28	0,58
Bulk density, g/cm^3	1,000	1,043	1,049	1,085	1,140	1,151	1,170	1,175	1,241

The table shows that the bulk density of the powder and content of oxygen is a function of the hydrogen humidity. The drier the hydrogen was that entered the recovery furnace, the lower the oxygen content and the bulk density of the powder.

The effect of the temperature recovery on the reduction of the oxygen content and the bulk density of the molybdenum metal powder was studied in a two-zone muffle furnace. This furnace allows the recovery temperature to be raised to $150\text{-}200\text{ }^{\circ}\text{C}$, which is consistent with the technology used to produce the existing Mch grade molybdenum. A boat was filled with carefully characterized molybdenum dioxide, charged into the hot zone of the furnace and maintained for 6 hours. After cooling, the metal powder was discharged and screened through a 180 silk mesh. The oxygen content and bulk density were also analysed. The oxygen content in the metal powder decreased compared with that of the standard Mch grade powder from 0.3 to 0,1-0,08%. Raising the recovery temperature significantly changed the properties of the powder. Standard metallic Mch grade molybdenum powder generally consists of particles with an average diameter (according to Fisher) of 2-4 microns; after it was reconstituted in a two-zone furnace, the average diameter was 8-10 microns. The bulk density of these powders was $1,74\text{-}1,95\text{ g}/\text{cm}^3$.

The resulting batch of bars (from the coarse powder) was deformed during rotational forging and drawing and did not experience brittle fracture. At the same time, the high recovery temperature ($1050\text{-}1100\text{ }^{\circ}\text{C}$) and increasing recovery time contributed to the improved removal of the fusible impurities.



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V. CONCLUSION

The study of the influence of the hydrogen humidity and recovery temperature on the reduction of the oxygen content and bulk density of molybdenum powder enabled the development of process conditions to produce metallic molybdenum powder with an oxygen content of -0,08-0,1% and a bulk density of up to 1,74-1,95 g/cm³. These findings represent a significant improvement in the quality of the metal, particularly the oxygen content, which is one of the most harmful molybdenum impurities.

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