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## EDITED TRANSLATION

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PREPARATION OF THE CHROMIUM CARBIDES  $Cr_7C_3$  and  $Cr_23C_6$ 

By: M. Kh. Freyd and V. A. Suprunov

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PREPARATION OF THE CHROMIUM CARBIDES  ${\rm Cr}_7{\rm C}_3$  and  ${\rm Cr}_{23}{\rm C}_6$ 

M. Kh. Freyd and V. A. Suprunov

(Ivanovo Chemicotechnological Institute)

Chromium carbides are high-melting, hard, and noncorroding substances [1-3] whose presence and distribution in chrome-nickel steels is connected in a group of works [4-7] to the onset of a tendency to intercrystalline corrosion.

To study the electrochemical and corrosion properties of the carbide phases of chromium it is necessary to prepare them in a single-phase condition; this process was studied in detail in works [8-10].

This work presents a refinement of the technological processes for preparing chromium carbides by reducing chromium oxides with carbon and the direct synthesis of chromium and carbon in industrial hydrogen furniset.

## Experimental Part

Chromium carbide  $\operatorname{Cr}_7\operatorname{C}_3$  was produced according to the reaction

$$70r_20_3 + 270 + 20r_70_3 + 2100$$
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Chromium oxide (GOST [All-Union State Standard] 2012-54) and lamp black (GOST 7885-50), first roasted at 800° and 400°C respectively for

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two hours, were screened to a fraction not larger than 25 µm. An adjustment to the composition of the charge was inserted according to data in [11]. The charge was mixed in mechanical rollers with addition of a solution of dextrin calculated as 5 g to 100 g of the charge. The charge was dried at 110°C for six hours and was pressed into briquettes  $25 \times 20$  mm in size at a pressure of 300 kg/cm<sup>2</sup>; the charge was again dried for two hours at 110°C and was subjected to carbidization. The results are presented in Table 1. The briquettes were heated in TsEP-356 industrial hydrogen furnaces in a stream of purified and dried hydrogen and were maintained at a given temperature for one hour and then were cooled to room temperature in the furnace cooler. The obtained product was ground and subjected to a second roasting for 15 minutes at a given temperature. The prepared product was analyzed for the total content of carbon [11] and for free carbon [12]. Bound carbon was determined by the formula

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$$C_{\text{bou}} = \frac{C_{\text{tot}} - C_{\text{free}}}{100 - C_{\text{free}}} 100\%.$$

The chromium content was determined by the persulfate-silver method [11]. Through X-ray structural analysis (Table 4) it was established that of the four groups of carbides, only the third consists of singlephase structures. The carbides obtained at temperatures higher than the optimum (1300°C) contain  $\text{Cr}_3\text{C}_2$  carbides as admixtures, and carbides obtained at lower temperatures contain the lower carbide  $\text{Cr}_{23}\text{C}_6$  and underreduced chromium oxide. Table 1 presents the ratio of the product weight after reaction A to the calculated weight of the carbide E, by which it is possible to judge the completeness of the reaction.

For carbide groups 3 and 4, for which the ratic A/B is close to the theoretical, the content of  $C_{\rm free}$  is insignificant and the amount of  $C_{\rm bou}$  is close to the stoichlometric composition. The optimum values of these indicators appear in carbides of the third group.

The variable  $\operatorname{Cr}_{2,2}\mathbb{C}_n$  is derived according to the reaction

$$\mathbb{C} \mathbb{C} \mathbb{P}^{1} + \mathbb{C} \mathbb{C} \rightarrow \mathbb{C} \mathbb{P}^{1} \mathbb{P}^{1} \mathbb{P}^{1}$$

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lts of e	Briquett	before heating	Calcu 230,00 107,00 101,60 171,60
. Resu	ŧ	lemper- ature, °C	1100 1100 1100 1100
Table 1		<b>.</b>	-004

( 7 \* 2 Results of experiments on producing the chromium Table 2.

رو.		adameter phases	Cr.,Co, + Cr,C, Cr.,Co, + Cr,C, Cr.,Cc, + Cr,C, Cr.,Cc, + Cr,C, Cr.,Cc, + Cr,C, + Cr_5C, Cr.,Cc, + Cr,C, + Cr_5C,			
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. guing		<sup>C</sup> total	5,68 6,68 6,20 5,55 5,52 6,33			
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Previously prepared electrolytic enromium mixed with lamp black was pressed into 10 × 10 mm briquettes at a pressure of 200 kg/cm<sup>2</sup> and was subjected to carbidization at different temperatures (see Table 2). The rise in temperature from 1000°C to the starting point was accomplished in 15 minutes and at the given temperature aging was accomplished for the same amount of time. From a charge of the same composition containing 20% less carbon than calculated, 5 groups of  $Cr_{23}C_6$  carbide were obtained. X-ray structural analysis fixed a single-phase structure in only the fourth group. In the remaining groups several carbide phases were detected. This is explained by the fact that the area where  $Cr_7C_3$  carbides exist is substantially wider than that for  $Cr_{23}C_6$ . The free energy of formation of  $Cr_7C_3$  is 2.5 times higher than for  $Cr_{23}C_6$  carbide (see the figure) [13]; at temperatures lower or higher than the optimum (1400°C), the reaction of  $Cr_7C_3$  formation suppresses the  $Cr_{23}C_6$  reaction.



Figure. Change in the free energy of formation  $\Delta F$  of the carbides  $Cr_{23}C_6$  (1) and  $Cr_7C_3$  (2) as a function of carbidization temperature.

The ratio A/B (see Table 2) changes little in the 1200-1500°C range and it approaches unity as the quantity of  $Cr_{23}C_6$  carbide in the product increases.

The content of carbon in carbides obtained at 1200-1350°C is greater than the stoichiometric, since the product contains mixtures of carbides which are enriched with carbon as compared to  $\text{Cr}_{23}\text{C}_6$  carbide. As the temperature of carbide synthesis is increased up to the optimum the content of bound carbon decreases and at the optimum temperature (1400°C) reaches its minimum, which corresponds to the stoichiometric composition of the  $\text{Cr}_{23}\text{C}_6$  carbide.

An increase in the sintering temperature leads anew to an increase in the amount of bound carbon, in connection with the formation of nighter samples.

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## Conclusions

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1. A two-stage process is proposed for obtaining the single-phase carbide  $\operatorname{Cr}_7C_3$  by reducing chromium oxide with lamp black in a TsEP-356 industrial hydrogen furnace. It consists of heating the charge in an atmosphere of hydrogen to 1300°C for 15 minutes, maintaining it at that temperature for one hour, cooling the product to room temperature with subsequent crushing and supplementary heating in a hydrogen atmosphere for 20 minutes.

2. The single-phase carbide  $\operatorname{Cr}_{23}\operatorname{C}_6$ , close in stoichiometric composition to the calculated, may be obtained in a stream of hydrogen by synthesis of metallic chromium and lamp black at 1400°C; the time of the temperature rise to 1400°C is 15 minutes and holding at the optimum temperature is for the same length of time.

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