CHROMIC OXIDE AS REFRACTORY MATERIAL

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The intensification of the thermal processes in the metallurgical and other industries makes it necessary to use new superduty refractory materials. Refractory oxides and their compounds will remain the principal materials for the foreseeable future [1]. Among the new oxide materials, chromic oxide, with its high melting point, is a promising type. Refractoriness, however, is not the only criterion of the suitability of a refractory, although it is an essential one. A refractory material must possess certain specific properties depending on the conditions of its service.

Chromic oxide has a corundumlike structure. In its natural form it is known as eskolaite, a mineral in which some of the Cr is substituted by V^{3+} and Fe^{3+}. Eskolaite may occur in chromites in the form of a decomposition product of a solid solution.

The most important chromium ore is chromite. Chromium occurs also in red lead ore, in which it was first discovered in 1797, but it was soon afterwards found also in chromite.

Chromium forms several oxides with oxygen. The stability of compounds of bivalent chromium is low; that of derivatives of trivalent chromium is higher, while the derivatives of hexavalent chromium are stable. Chromium is tetravalent or pentavalent in only a few compounds and monovalent in still fewer.

The Cr–O system can be divided into two systems in accordance with the oxygen content, viz., $Cr-Cr_2O_3$ with a low, and $Cr_2O_3-CrO_3$ with a high, oxygen content. The former comprises the compounds which are stable at high temperatures; the latter, the compounds which are stable at relatively low temperatures.

Chromium and Cr_2O_3 coexist at 780-1300°C without appreciable mutual solubility. The solubility of the oxygen in solid chromium at 1350°C is 0.1 at. % and decreases with the temperature. The $Cr-Cr_2O_3$ system was found to contain a phase in the form of chromous oxide CrO, which on heating to 967°C decomposes and gives Cr_2O_3. However, this is not the peak temperature at which chromous oxide can exist, i.e., it is often found in molten slag. Experiments have shown that at 1500°C CrO undergoes disproportionation to chromic oxide and chromium. The composition of the eutectic (20% Cr, 80% Cr_2O_3) with melting point 1660°C in the $Cr-Cr_2O_3$ system is very similar to the molecular composition of CrO.

The phase Cr_3O_4 is stable only at very high temperatures; at temperatures below the liquidus curve it undergoes disproportionation to Cr_2O_3 and CrO.

Chromous oxide thus exists in the $Cr-Cr_2O_3$ system in every case in the form of a diatomic gaseous molecule. It may occur in significant quantities in slags that have interacted with a chromous metal by analogy with silica, which is metastable in the solid phase and stable in molten slag. For example, in melts of the $CaO-MgO-Al_2O_3-Cr_2O_3-SiO_2$ system a large proportion of the chromium occurs in the form of its lower oxide. The proportion of bivalent chromium increases with the degree of reduction of the melt.

The content of chromous oxide in the melt increases with a reduction in the amount of ferrous oxide, i.e., chromous and ferrous oxides are antagonistic. Bivalent chromium occurs in appreciable amounts in acid slags, and its proportion decreases with an increase in the basicity of the slag.

The content of chromous oxide in chromium slag depends on the material with which the slag is in contact. The contact zone of chromium slag with steel contains CrO as well as FeO and MnO. The contact zone of the slag with air contains Cr_2O_3 (Fe_2O_3, Mn_2O_3); consequently, CrO and Cr_2O_3 are present in chromium slag [2].

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TABLE 1. Characteristics of Chromic Oxides [3]

Oxide	Mol. wt., g/mole	Mol. vol., cm³/mole	Density, g/cm³	Melting point (M) or decom. point (D), °C
Cr_3O	171,99	28,06	6,13	—
CrO	68,00	10,53	6,46	1727*M
Cr_3O_4	219,99	42,89	5,13	—
α-Cr_2O_3†	151,99	28,18	5,21	2330 M
CrO_2	83,99	17,15	4,90	477 D
Cr_5O_{12}	451,97	121,82	3,71	547 D
Cr_8O_{21}	751,96	244,94	3,07	367 D
$CrO_{2,906}$	98,49	34,20	2,88	237—277‡
CrO_3	99,99	35,46	2,82	188 M

*According to [6] the melting point of CrO is 1640°C.

† Eskolaite.

‡ The interval of existence.

The existence of an individual solid phase of CrO has not been substantiated, whereas Cr_3O_4 may be a stable high-temperature phase. Orthosilicate of bivalent chromium Cr_2SiO_4 is a known compound, on the other hand, and forms a solid solution with forsterite.

It is known that a cubic system of the oxide Cr_3O exists, but it has not been isolated in its pure form. It is formed by reducing chromic oxide with metallic titanium, vanadium, and zirconium and by oxidizing chromium powder with water vapor at 600°C in the presence of a large amount of hydrogen [3, pp. 64-68; 4, pp. 5-24; 5, pp. 241-254].

Chromic oxide is the basic phase and is of interest as refractory material. When heated in air, metallic chromium burns rapidly and forms chromic oxide, which is insoluble in water and acids. Chromic oxide is a green or dark-green powder; when produced at a high temperature it has a metallic glitter and is black, but it turns green when the powder is triturated.

With regard to the melting point of chromic oxide, opinion is divided, possibly because it varies as a result of the partial reduction of Cr^{3+} to Cr^{2+}, which lowers the melting point. The melting point appears to lie between 2265 and 2330°C.

The true theoretical density of Cr_2O_3 is 5.23 g/cm³, while the density of the synthesized mineral is 5.215 g/cm³; the microhardness,* about 3200 kgf/mm² and the Mohs hardness, 8.5; the syngony is hexagonal and the mean index of refraction, 2.50; chromic oxide contains 68.42 wt.% Cr and 31.58 wt.% O_2.

The specific heat of chromic oxide at 25°C is 28.38 cal/mole • deg C. The coefficient of linear expansion over 20-1400°C is $9.6 \cdot 10^{-6}$ °C^{-1}.

In combination with the oxides of a large number of other metals, chromic oxide gives a compound of the type of spinel $MeO \cdot Cr_2O_3$.

The $Cr_2O_3-CrO_3$ system comprises a large number of known phases (Table 1).

Heating causes CrO_3 to decompose; the composition of the decomposition products depends on the temperature and pressure. The valence of the chromic oxide decreases with an increase in the temperature.

Heating of CrO_3 at standard pressure gives $CrO_{2.906}$ at 237°C; at a temperature slightly above 250°C this oxide is decomposed to Cr_8O_{21} and oxygen.

When CrO_3 is heated to 275°C it forms Cr_8O_{21}, which decomposes at 367°C and gives Cr_5O_{12} and oxygen. A further increase in the heating temperature of CrO_3 results in the formation of Cr_5O_{12} as the decomposition product; this oxide decomposes at 547°C to Cr_2O_3 and oxygen.

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*Vickers pyramid.

TABLE 2. Binary Systems with Chromic Oxide[*]

Second component in system[†]	Binary compound in system[†]	Eutectics of system	Approx. Cr_2O_3 content in eutectics, wt. %	mp of the eutectic, °C
CaO(2625)	$CaCr_2O_4$ (2170)	Cr_2O_3—$CaCr_2O_4$ $CaCr_2O_4$—CaO	85 50	2100 1930
MgO (2825)	$MgCr_2O_4$ (2400)	Cr_2O_3—$MgCr_2O_4$ $MgCr_2O_4$— solid solu- of MgO with $MgCr_2O_4$	80 [‡] 33 [‡]	2090 2350
Fe_2O_3 (1730)	Continuous series of solid solns. (Fe, Cr)$_2$O$_3$	—	—	—
Al_2O_3 (2045)	The same (Al, Cr)$_2$O$_3$	—	—	—
ZrO_2 (2700)	None	Cr_2O_3—ZrO_2	66	2090
TiO_2 (1870)	$Cr_2Ti_2O_7$ (1980)	Cr_2O_3—$Cr_2Ti_2O_7$ $Cr_2Ti_2O_7$—TiO_2	64 4 [‡]	1880 1760
SiO_2 (1728)	None	Two immiscible liquids containing 33-90 mole %SiO_2	—	2200

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[*] The melting point of chromic oxide is 2330°C.

[†] The mp, °C, is stated in parentheses.

[‡] Stated content in mole %.

The heating of CrO_3 to a moderate temperature (400-500°C) at a high pressure (200-600 atm or higher) results in the formation of CrO_2. This oxide has a region of homogeneity within the composition limits $Cr_{1.88-2.18}$. In air at 477°C or higher CrO_2 decomposes to Cr_2O_3 and oxygen [3, pp. 64-68; 7].

The heating of CrO_3 thus results in the formation of a series of oxides which continue to decompose with a further increase in the temperature and in the end form chromic oxide.

Chromium trioxide CrO_3 is the anhydride of chromic acid. Its color is dark red, and it is readily dissolved in water to give chromic (H_2CrO_4) and dichromic ($H_2Cr_2O_7$) acid, which are known only in solution. Chromic trioxide is highly toxic and a very potent oxidizer.

The explanation lies in the fact that chromium trioxide, which melts at 180-202°C, decomposes at an increasing rate with an increase in the temperature and gives off oxygen. Moreover, CrO_3 can exist also in the gaseous state.

At room temperature or when lightly heated CrO_3 is almost stable in air because crystallochemical conversions will not readily develop at a low temperature.

Chromic oxide is therefore the only near-stable oxide in the Cr—O system at temperatures below 1600°C and oxygen pressures below 1 technical atm.

In highly oxidizing conditions chromic oxide may dissolve excess oxygen but chromic oxide of a nonstoichiometric composition cannot be produced at a pressure above 1 technical atm [8, pp. 127-130].

Chromic oxides are therefore sensitive to changes in the composition of the environment and in the temperature. A characteristic feature of chromic oxide is that it reacts both with oxygen and with gaseous or solid reducing agents, a fact which somewhat limits the scope for using chromic oxide refractories.

Of considerable significance for the use of chromic oxide as a refractory are the characteristics of the systems which it forms with CaO, MgO, FeO, Fe_2O_3, Fe_3O_4, Al_2O_3, TiO_2, ZrO_2, and SiO_2.

The data in Table 2, which were compiled from [3, pp. 99-101, 118-120, 135-136, 140-144], show that all binary compounds in binary systems with chromic oxide have a high melting point.

In nonoxidizing conditions calcium chromite $CaCr_2O_4$ forms in the system with CaO and undergoes $\beta \rightarrow \alpha$ modification at 1570°C with a simultaneous 3% increase in volume. In oxidizing conditions at a low temperature, not only calcium chromite is formed but also calcium chromate $CaCrO_4$ as a result of the oxidation of the calcium chromite; this process is accompanied by a large increase in volume. The chromate is not stable and decomposes at 1060°C.

Unlike calcium chromite, the magnesiochromite $MgCr_2O_4$ in the Cr_2O_3-MgO system is stable during heating in air. Magnesium chromate $MgCrO_4$ decomposes at 525-645°C and forms magnesiochromite.

The $Fe_2O_3-Cr_2O_3$ system forms a continuous series of solid solutions of the oxides. At high temperatures the Fe_2O_3 dissociates and the system is converted to the ternary type $FeO-Fe_2O_3-Cr_2O_3$.

A continuous series of solid solutions of the oxides forms also in the system of Cr_2O_3 with Al_2O_3. A discontinuity occurs at temperatures below 800°C.

The system Cr_2O_3 with ZrO_2 is the simplest one with a high-temperature eutectic [3, p. 141; 6, pp. 466-467].

With Cr_2O_3 the compound $Cr_2Ti_2O_7$ in the $Cr_2O_3-TiO_2$ system forms a high-temperature eutectic at a temperature above 1200°C.

The limiting solubility of titanium oxides with chromic oxide at 1300-1900°C is 5 mole %. Titanium ions are present in a solution in the tetravalent and trivalent state, which gives a nonstoichiometry which is probably of the type of cation vacancies. With up to 20 mole % excess TiO_2 a solid solution is formed together with chromium titanate Cr_2TiO_5, and with up to 50 mole % TiO_2 chromium dititanate $Cr_2Ti_2O_7$ is formed as well as titanate [9].

No compounds or solid solutions are formed in the $Cr_2O_3-SiO_2$ system. The region of liquation in this system is large. Over the temperature range 1720-2200°C solid chromic oxide and a solution exist side by side, but above 2200°C the system forms two solutions [3, pp. 143-144; 6, pp. 112-114].

Two-component systems with Cr_2O_3 are highly refractory because the melting point of the binary compounds in these systems lies within 1980-2400°C and the binary eutectic within 1760-2350°C.

A summary of the ultimate triangles in ternary systems with chromic oxide is given below [3]. Note that most of these systems have not been investigated in depth.

System with chromic oxide	Ultimate triangles
CaO, MgO	$CaCr_2O_4$—Cr_2O_3—$MgCr_2O_4$
CaO, FeO	$CaCr_2O_4$—Cr_2O_3—$FeCr_2O_4$
CaO, Fe_2O_3	$CaCr_2O_4$—Cr_2O_3—Fe_2O_3
CaO, Al_2O_3	$CaCr_2O_4$—Cr_2O_3—$CaAl_{12}O_{19}$; $CaAl_{12}O_{19}$—Cr_2O_3—Al_2O_3
CaO, SiO_2	CaSiO—Cr_2O_3—SiO_2; $Ca_3Si_2O_7$—Cr_2O_3—$CaSiO_3$; Ca_2SiO_4—Cr_2O_3—$Ca_3Si_2O_7$; Ca_2SiO_4—Cr_2O_3—$CaCr_2O_4$; Ca_2SiO_4—$CaCr_2O_4$—Ca_3SiO_5; Ca_3SiO_5—$CaCr_2O_4$—CaO
CaO, ZrO_2	$CaCr_2O_4$—Cr_2O_3—ZrO_2
CaO, TiO_2	$CaTiO_3$—Cr_2O_3—$Cr_2Ti_2O_7$; $CaTiO_3$—Cr_2O_3—$CaCr_2O_4$
MgO, FeO	$FeCr_2O_4$—Cr_2O_3—$MgCr_2O_4$
MgO, Fe_2O_3	$MgCr_2O_4$—Cr_2O_3—Fe_2O_3
MgO, Al_2O_3	$MgAl_2O_4$—Cr_2O_3—Al_2O_3; $MgAl_2O_4$—Cr_2O_3—$MgCr_2O_4$
MgO, SiO_2	$MgSiO_3$—Cr_2O_3—SiO_2; $MgSiO_3$—Cr_2O_3—$MgCr_2O_4$
MgO, ZrO_2	$MgCr_2O_4$—Cr_2O_3—ZrO_2
MgO, TiO_2	$MgCr_2O_4$—Cr_2O_3—$Cr_2Ti_2O_7$
FeO, SiO_2	$FeCr_2O_4$—Cr_2O_3—SiO_2
FeO, TiO_2	$FeCr_2O_4$—Cr_2O_3—$Cr_2Ti_2O_7$
FeO, ZrO_2	$FeCr_2O_4$—Cr_2O_3—ZrO_2; $FeCr_2O_4$—ZrO_2—FeO
FeO, Fe_2O_3	$FeCr_2O_4$—Cr_2O_3—Fe_2O_3
Fe_2O_3, Al_2O_3	Fe_2O_3—Cr_2O_3—$FeAlO_3$; $FeAlO_3$—Cr_2O_3—Al_2O_3
Fe_2O_3, SiO_2	Fe_2O_3—Cr_2O_3—SiO_2
Fe_2O_3, TiO_2	Fe_2O_3—Cr_2O_3—$Cr_2Ti_2O_7$
Fe_2O_3, ZrO_2	Fe_2O_3—Cr_2O_3—ZrO_2
Al_2O_3, SiO_2	Al_2O_3—Cr_2O_3—$Al_6Si_2O_{13}$; SiO_2—Cr_2O_3—$Al_8Si_2O_{13}$
Al_2O_3, TiO_2	$Cr_2Ti_2O_7$—Cr_2O_3—Al_2TiO_5; Al_2O_3—Cr_2O_3—Al_2TiO_5
Al_2O_3, ZrO_2	Al_2O_3—Cr_2O_3—ZrO_2
ZrO_2, SiO_2	$ZrSiO_4$—Cr_2O_3—SiO_2; $ZrSiO_4$—Cr_2O_3—ZrO_2
ZrO_2, TiO_2	$Cr_2Ti_2O_7$—Cr_2O_3—ZrO_2
TiO_2, SiO_2	$Cr_2Ti_2O_7$—TiO_2—SiO_2; $Cr_2Ti_2O_7$—Cr_2O_3—SiO_2

In the ternary systems considered here the chromic oxide coexists with other oxides and their binary compounds because ternary compounds cannot form with chromic oxide.

Systems containing silica are characterized by a large region of liquation.

Ternary eutectics have been determined in the following systems: $CaO-Cr_2O_3-SiO_2$ [containing 3 (1418°C) and 7 (1407°C) wt. % Cr_2O_3]; $Mg-Cr_2O_3-SiO_2$; $MgO-Cr_2O_3-ZrO_2$ (1860°C); $FeO-Cr_2O_3-SiO_2$ (1155°C); $FeO-Cr_2O_3-ZrO_2$ (1325°C); $Al_2O_3-Cr_2O_3-SiO_2$ (1580°C, 1 wt.% Cr_2O_3); and $Cr_2O_3-TiO_2-SiO_2$ (1527°C). Thus the known ternary eutectics occur mainly in systems with silica and in some with zirconia.

The melting temperature of the eutectic is lowest in systems with ferrous oxide together with SiO_2 or ZrO_2.

Ternary solid solutions* were found to occur in many of the systems concerned here. The $CaO-Fe_2O_3-Cr_2O_3$ system contains several s.l. Ca $(Cr, Fe)_2O_4$ which occur also in oxidizing conditions in the $CaO-FeO-Cr_2O_3$ system.

In the $CaO-Al_2O_3-Cr_2O_3$ system the compound $CaAl_2O_4$ dissolves readily in $CaCr_2O_4$, but not vice versa. Ternary s.l. form in air in the $CaO-Cr_2O_3-ZrO_2$ system accompanied by partial oxidation of the Cr_2O_3.

Limited s.l. have been found to exist in the $CaO-Cr_2O_3-TiO_2$ system.

At a low oxygen partial pressure the $MgO-FeO-Cr_2O_3$ system contains s.l. of spinels $(Mg, Fe)Cr_2O_4$.

The $MgO-Fe_2O_3-Cr_2O_3$ system can form s.l. $Mg(Fe, Cr)_2O_4$, while s.l. $Mg(Al, Cr)_2O_4$ with mp 510°C form in the $MgO-Al_2O_3-Cr_2O_3$ system.

In $MgO-Cr_2O_3-Si_2O_2$ and $MgO-Cr_2O_3-ZrO_2$ systems the s.l. occupy a small region, while in the $MgO-Cr_2O_3-TiO_2$ system a continues series of s.l. is formed between the phases $MgCr_2O_4$ and Mg_2TiO_4, and limited s.l. between $MgTi_2O_5$ and Cr_2TiO_5.

In ternary systems that contain FeO and Cr_2O_3, s.l. are formed if the third component is TiO_2 (between $FeTi_2O_5$ and Cr_2TiO_5) or ZrO_2 (very limited ones) but no s.l. are formed when the third component is SiO_2.

A continuous series of spinel s.l. of Fe_3O_4 and $FeCr_2O_4$ which do not decompose on cooling was determined in the $FeO-Fe_2O_3-Cr_2O_3$ system.

The s.l. determined in the $Fe_2O_3-Cr_2O_3-Al_2O_3$ system cover an extensive range and decompose at about 1500°C and when the proportion of Cr_2O_3 is smaller than 30 wt.%. The region of ternary s.l. contracts with a decrease in the temperature.

The $Fe_2O_3-Cr_2O_3-TiO_2$ system contains very numerous s.l. In the phase $Cr_2Ti_2O_7$ two or three chromium atoms may be substituted by iron atoms; in phase $Fe_4Ti_3O_{12}$ up to 15 mole % of the Fe_2O_3 is substituted by Cr_2O_3.

In the $Al_2O_3-Cr_2O_3-SiO_2$ system limited s.l. of chromic oxide (up to 10 wt.% Cr_2O_3) are formed in mullite at 1600°C.

Limited s.l. are formed also between Al_2TiO_5 and $Cr_2Ti_2O_7$ in the $Al_2O_3-Cr_2O_3-TiO_2$ system. The limiting solubility of the second compound in the first does not exceed 6-7 wt.%, while that of the first in the second is about 35 wt.% [10].

In the $Cr_2O_3-ZrO_2-SiO_2$ system the s.l. form very small regions, one of which adjoins the $Cr_2O_3-ZrO_2$ side near angle Cr_2O_3 and another adjoins the SiO_2-ZrO_2 side at angle ZrO_2 [3, p. 236; 11, pp. 294-295].

CONCLUSIONS

The article deals with chromic oxide and other oxides of chromium and with the systems which chromic oxide forms with other oxides and which are of importance in the production and application of refractories. In most systems Cr_2O_3 coexists with the other oxides and their high-melting compounds.

Ternary compounds with chromic oxide are absent in these systems, and ternary eutectics are rare; the binary eutectics are characterized by high melting points. The systems contain large regions of solid solutions between the oxides and their highly refractory compounds.

These are the factors which render chromic oxide a promising material for the production of superduty refractories.

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*The term "solid solution(s)" is henceforth abbreviated to s.l.

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LIGHTWEIGHT FOAMED FILLER IN GRANULAR FORM FROM RAW KAOLIN

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Lightweight refractories with good strength and heat-insulation properties can be produced by the foaming method from lean fine-dispersion aluminosilicate compositions with a stabilizing additive [1, 2].

Experiments carried out at the Ukrainian Institute of Refractories provided evidence that this method can be used with good results also for the production of lightweight aluminosilicate filler of low apparent density in a production cycle of 2.5-3.0 h in place of 3 or 4 days.

The present writers carried out laboratory-scale technological investigations and experiments in the production of a lightweight filler in granular form from secondary kaolin by the foam method.

The granules are used for the manufacture of heat-insulation concrete, ramming compounds, fired and unfired lightweight products, and heat-insulation filler.

In contrast to the known production technology for lightweight refractories from a foamed lean composition, the lightweight granular filler is produced from foamed kaolin clinker without added grog by a method which includes a stage in which the foamed composition is subjected to a high-speed heat treatment.

In the proposed method [3] a slip is prepared from kaolin and converted to stable foam which is next plasticized and then coagulated and granulated by rapid high-temperature drying after which the granules are subjected to high-speed firing.

The composition is plasticized by adding aluminum sulfate or alum. The hydrolysis of these salts produces a change in the pH of the medium and results in the formation of colloidal aluminum hydroxide, which is

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