THEORY AND TECHNOLOGY OF SINTERING, HEAT,
AND CHEMICAL HEAT-TREATMENT PROCESSES

STRUCTURING ON SINTERING IN THE PRESENCE
OF LIQUID PHASE IN Cr – Cu – IRON GROUP METAL
SYSTEMS. I. Cr – Cu SYSTEM

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Structuring has been examined for Cr – Cu composites under conditions of impregnation and subsequent liquid-phase sintering at 1200 °C in a vacuum of (2-4) · 10⁻³ Pa with reduced and electrolytic chromium powders. The size distribution for the particles of the refractory component in the microstructure containing the reduced chromium on liquid-phase sintering for 60 min corresponds to a logarithmic normal distribution; the distribution parameters are sensitive to the volume fraction of refractory particles. The calculated values for the dihedral angle are close to one of the modes of the distribution for the dihedral angles in the microstructure for specimens made of electrolytic chromium (115 °). At 1200 °C, the equilibrium Crₙ – Cul system obeys the condition \( \frac{\sigma_{sl}}{\sigma_{ss}} \geq 0.5 \). This indicates the probability of formation or preservation of framework structure elements during the liquid-phase sintering, which are observed by experiment in specimens containing reduced chromium.

Keywords: composite, liquid-phase sintering, chromium, copper, iron-family metal, surface energy, dihedral angle, framework structure, matrix structure.

INTRODUCTION

System Cr – Cu composites are used [1] in electrical contact materials employed in vacuum switching equipment for medium voltages (10-36 kV) and high currents (over 10 kA). The working characteristics are dependent on the relation between the properties of the refractory and fusible components, the strength of the bonds at the boundaries between them, and microstructure features [2]. Improvements to the technology of making the Cr – Cu materials by means of powder metallurgy require knowledge of the regularities in the structure formation on sintering in the presence of liquid phase (liquid-phase sintering, LPS).

Good adhesion is required between the refractory component and the liquid, which is attained in particular by alloying [3] and is a necessary condition for metallic contact and maximum strength in the interphase boundaries. The purpose of the present study is to examine the effects of added Fe, Co, and Ni on the microstructure formation in Cr – Cu composites in the impregnation with copper for various chromium powders under vacuum followed by LPS at 1200 °C. In the present paper, we present results for specimens made by impregnating chromium with copper without additives.
Fig. 1. Shapes of powder particles for reduced chromium (a) and microstructures of specimens obtained by impregnating reduced chromium with copper and subsequent LPS for 3 min (b) and 30 min (c); volume proportion of refractory particles $V = 40\%$

**MATERIALS AND METHODS**

We used two forms of chromium powder: as reduced by calcium hydride and electrolytic with mean particle sizes of 8 and 300 $\mu$m, respectively. The mass fractions in % of impurities in the reduced chromium powder were: 0.15 O, <0.007 N, <0.003 H, 0.06 C, 0.09 Si, 0.15 Fe, 0.1 Ca, 0.1 Ni; in the electrolytic chromium they were: 0.04 O, <0.007 N, <0.003 H, 0.008 C, 0.008 Si, 0.002 S, <0.001 P, 0.008 Fe, 0.005 Ni.

The porous blanks made of freely poured chromium powder were impregnated under vacuum* with molten copper (99.999% Cu) at 1200°C (with a mass ratio of chromium and copper 1 : 1 or excess liquid). The times for the subsequent LPS (at 1200°C) varied from 3 min to 2.5 h. On isothermal hold for 3 min, we used separate heating of the fusible and refractory components: when the working temperature was attained, the fusible component was placed on to the chromium blank, and the contact time was determined from the instant of the copper melting. The methods provided good adhesion and complete impregnation. The microstructures of the sintered specimens were examined on sections prepared in the standard way for qualitative and quantitative metallography (after etching the sections in 40% HCl). The microstructure was analyzed quantitatively by means of a computer program: “SIAMS-600 industrial system for processing and analyzing images” (http://siams.com). The linear dimensions of the refractory particles were estimated by calculating the Feret diameter $F_{\text{avg}}$ [4] by averaging the sizes of projections of the particles in 64 directions. The data sample for each specimen included 250-400 sections of particles on 10-15 fields of view. The experimental values of the dihedral angle (at the boundary between two adjacent grains of refractory phase in contact with the grain of fusible phase) were determined by Parker’s method [5]. Those angles were measured on processing optical images of the sections by means of the “AllPlan-2003” software package (“Nemetchek AG”), with the sample volume constituting 316 measurements, error of measurement not exceeding 0.5 deg.

**DISCUSSION**

*Microstructures of Cr – Cu composites made with reduced chromium. Refractory-particle size distribution.* Figure 1a shows a typical clump of particles for the initial chromium powder reduced with calcium hydride. It consists of intergrown closed chains of particles with prominent facets (scanning electron microscope image).

When the copper had impregnated the chromium powder, the volume fraction of the refractory component was $V = 42 \pm 4\%$ (with excess liquid during the impregnation) or $V = 56 \pm 4\%$ (with equal masses of chromium and copper). After the impregnation** (including with excess liquid), the microstructure showed groups of particles characteristic of the initial chromium powder (Fig. 1b), i.e. in the LPS, the structure elements of the initial chromium powder were preserved and/or new contacts arose between them (Fig. 1b and c, and Fig. 2a).

* Without breaking the sealing on the vacuum chamber (in each experiment), the chromium powder was first outgassed by heating in vacuum (between than (2-4) $\cdot 10^{-3}$ Pa) to 900°C; the refractory was an Al$_2$O$_3$ crucible.

** Isothermal hold for 3 min (after impregnation) was taken as the initial moment in the formation of the microstructure during LPS.