

Title	Determination of Density Change of Glass by the Sink-Float Method. (VI) : Change of Density of Glass with Time at Constant Temperature
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The X-ray analysis of metallic copper specimens which were deposited on the surface of zinc clod immerged in the above solution by the difference of the electrolytical solutional tension, was performed by the Laue method, utilizing the heterogenous X-rays emitted from copper anticathode.

The results thus obtained can be summarised as follows:

(1) Metallic copper deposited from cupric sulphate to which HCl or CH₃COOH was added, were composed of perfectly irregular aggregation of micro-crystals of the diameter 10⁻³~10⁻⁴cm. The higher the acid concentration, the more the porous copper became. When the added constituent was H₂SO₄, porous copper deposited was composed of comparatively large microcrystals of the diameter 10⁻²~10⁻³cm.

(2) In the case of addition of sulphates to cupric sulphate solutions, copper closely deposited on the surface of Zinc clod, having some special germs.

The deposited ground copper was composed of irregular aggregations of micro-crystals of 10⁻²cm. But the germinal copper had fibre structure having one or two axes <110>.

Laue photograph showed that this fibre structure became clear in the order of Na₂SO₄, ZnSO₄ and NaHSO₄.

(3) Quantity of sulphate added had no influence on the fibre structure, when the concentration of cupric sulphate was constant (18 or 28 percent in weight), while in the case of the constant quantity of sulphate was added, the higher the concentration of the cupric sulphate, the clearer the fibre structure became.

(4) Usually copper deposited from the above solution contained reddish-black Cu₂S, but copper from Fehling's solution did not contain such compound and had some copper germs which might have <110> fibre axis.

10. Determination of Density Change of Glass by the Sink-Float Method. (VI)

Change of Density of Glass with Time at Constant Temperature

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The flotation method of varying the temperature of bath at a constant rate, which has been used successfully to determine small variations in glass density for chemical composition control in factory practice, was proved to be applicable for studying the configurational change of glass during heat treatment in the annealing range (this Bull, 20 54, 1950; 24 71, 25 62, 26 70, 1951).

For the density measurement, rod samples of a soda-lime glass (*ibid.* 19 52, 1949) were treated for various times in a laboratory furnace kept at constant

temperatures and rapidly cooled down in air to room temperature. As examples, the changes in density with time are shown in Table 1-a, b and c.

Table 1. Change in Density (g./cm³.) at Room Temperature with Time of Heat Treatment.

(a) 540°C			(b) 530°C			(c) 460°C		
Time (min.)	Density Increase	Density Decrease	Time (min.)	Density Increase	Density Decrease	Time (hr.)	Density Increase	Density Decrease
0	2.4887 (1)	2.4975 (2)	0	2.4887 (1)	2.4975 (2)	0	2.4887 (1)	2.5003 (3)
2.5	2.4918	2.4958	2.5	2.4916	2.4966	2.5	2.4961	2.5003
5	2.4924	2.4949	5	2.4926	2.4964	5	2.4971	2.5004
10	2.4927	2.4936	10	2.4935	2.4957	10	2.4983	2.5005
20	2.4928	2.4928	20	2.4938	2.4951	20	2.4992	2.5006
			40	2.4939	2.4948	40	2.5000	2.5013
			80	2.4941	2.4944	80	2.5010	2.5017
			120	2.4942	2.4942	280	2.5023	2.5020

(1): Density of rod-as-drawn, 4mm. in diameter. (2) and (3): Density of samples treated at 480°C for 4hrs. and 10 days respectively.

From the above figures, it is shown that the change in density, D , with time, t , can not be represented by the relaxation equation of Maxwell type: $d(D_r - D)/dt = k_1 \cdot (D_r - D)$, where D_r is equilibrium density at temperature $T^\circ\text{C}$, nor of Adams-Williamson type: $d(D_r - D)/dt = k_2 \cdot (D_r - D)^2$, as the rate of change in density is extremely rapid while the difference, $(D_r - D)$, is large and the rate decreases rapidly as D approaches to D_r . To interpret these figures, it seems important to take account of the change in viscosity of glass with time at constant temperatures in the annealing range.

11. Factors Affecting Diffusion Velocity of Oxygen through Heat-Resistant Enamel Coatings

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At high temperatures, oxygen in air diffuses through an enamel layer and reacts with the base metal. Therefore, the property to prevent the diffusion of oxygen is most essential for the heat-resistant enamel. In order to acquire fundamental knowledge about factors which affect the diffusion velocity of oxygen, the authors measured successively the volume of oxygen, which diffuses through an enamel layer during heating at 800°C. The method used for the measurement is the same as reported by one of the authors (Jap. Ceram. Assoc. 1949, 57, 96). With a few exception, the frits now generally applied as the heat-resistant coatings