# MIKROSKOPIE

ZENTRALBLATT FÜR MIKROSKOPISCHE FORSCHUNG UND METHODIK

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## SOME PROBLEMS OF POWDER METALLURGY SOLVED BY THE MICROSCOPE

*With 8 figures*


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**(Sylvania Electric Products Inc., Bayside, N.Y.)**


Sintering, the central problem of powder technology, has attracted the attention of metallurgists and physicists for a good many years. The material particles in mutual contact are not in thermodynamical equilibrium because their surface free energy is not at a minimum. During heating those particles form common interfaces. This takes place in the case of metals by lattice or surface diffusion (1) and in the case of amorphous materials, such as glass or wax by viscous flow (2, 3). The experimental investigations of these processes were made possible by developing a special experimental technique which warrants a more detailed description.

In order to distinguish which of the above mentioned mechanisms is responsible for welding the particles together during sintering, we must measure the radius x of the formed interfaces as a function of time. If $x^5 \sim t$, where t is time of sintering at a given constant temperature, lattice diffusion is the actual mechanism; if $x^7 \sim t$, it is surface diffusion, and if $x^2 \sim t$ viscous flow occurs. The measurement of x can be carried out in the following way. The round metallic or glass particles are dispersed on a flat block of the same material. The surface of this block in the case of metals must be polished and cleaned by repeated etching. In the case of glass, degreasing is sufficient. Specimens prepared in this manner are then heated at a given temperature for the desired period of time. Metallic particles such as copper or nickel can be most satisfactorily heated in dry hydrogen or any neutral gas. Silver or glass particles may be heated in air. After heating the metallic blocks together with the particles sintered to them they are nickel plated in order not to break the bond between the particles and the base during polishing. They are then mounted in bakelite or lucite. The glass specimens are of course directly mounted in plastic. The mounted specimens are cut, polished, etched and examined under the microscope. The radii

of the interface can readily be measured either on the photomicrographs or directly with the micrometer eye-piece. The interface is circular, or nearly so; therefore in order to measure its true diameter, repeated polishing and measuring are necessary till the maximum diameter is obtained. Fig. 1, a, b, c, represents the progress of sintering of copper particles at 800° C. for 2, 4 and 16 hours. Fig. 2, a, b, c, represents the sintering of copper at 700° C. for the periods of 2, 8 and 41 hours. The black spots are holes left behind after deoxidizing copper with hydrogen. Fig. 3, a, b, c, shows the progress of sintering of 300 $\mu$ diameter silver particles at 800° C. for 6, 16 and 24 hours.

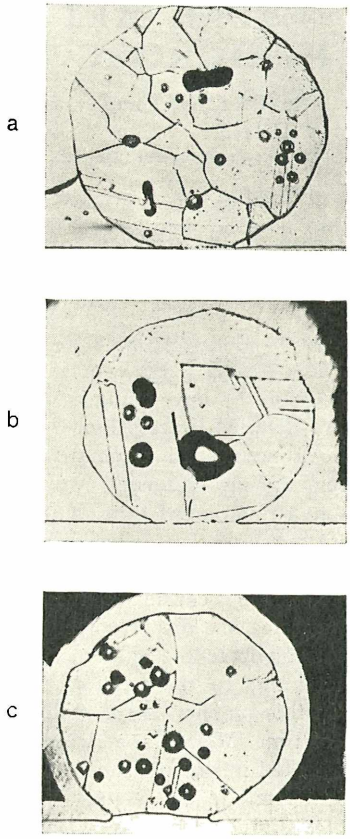



*Fig. 1. Copper Powder on Copper Block.*

*a = sintered at 800° C. for 2 hours. Mag. 900:1.*
*b = sintered at 800° C. for 4 hours. Mag. 1000:1.*
*c = sintered at 800° C. for 16 hours. Mag. 800:1.*

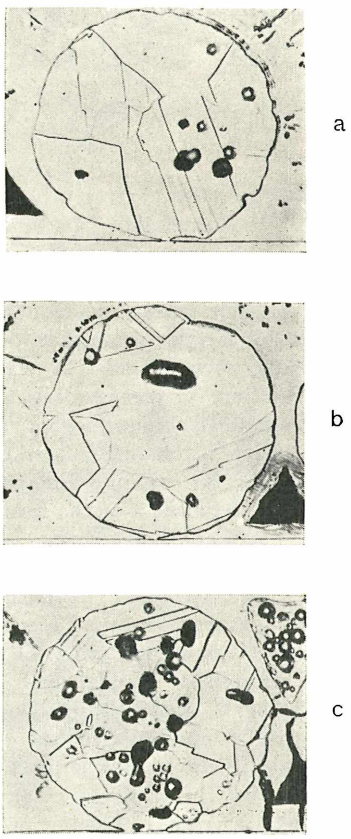
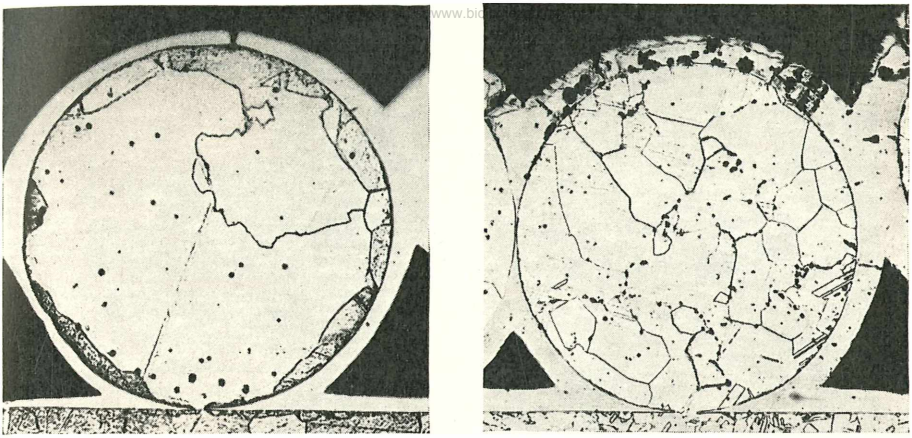
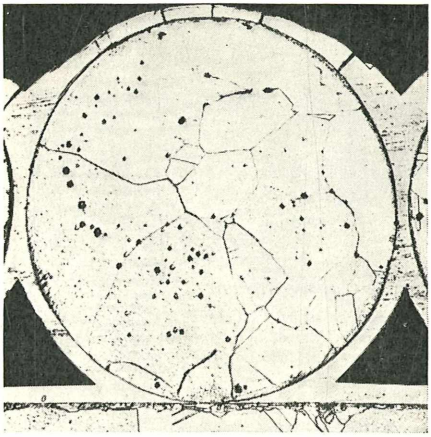



*Fig. 2. Copper Powder on Copper Block.*

*a = sintered at 700° C. for 2 hours. Mag. 800:1.*
*b = sintered at 700° C. for 8 hours. Mag. 900:1.*
*c = sintered at 700° C. for 41 hours. Mag. 900:1.*



a b



c

*Fig. 3. Silver Powder on Silver Block.*

*a = sintered at 800° C. for 6 hours. Mag. 250:1.*
*b = sintered at 800° C. for 16 hours. Mag. 250:1.*
*c = sintered at 800° C. for 24 hours. Mag. 250:1.*

Fig. 4, a, b, c, represents an advanced stage of sintering of copper particles heated at 1040° C. for 16½ hours. Fig. 5 gives two cross-sections through the mass of sintered unpressed copper powder heated at 800° C. for 6 hours.

Examination of these pictures reveals that the space between the particle and the flat surface of the metallic block is limited by the surface of rotation

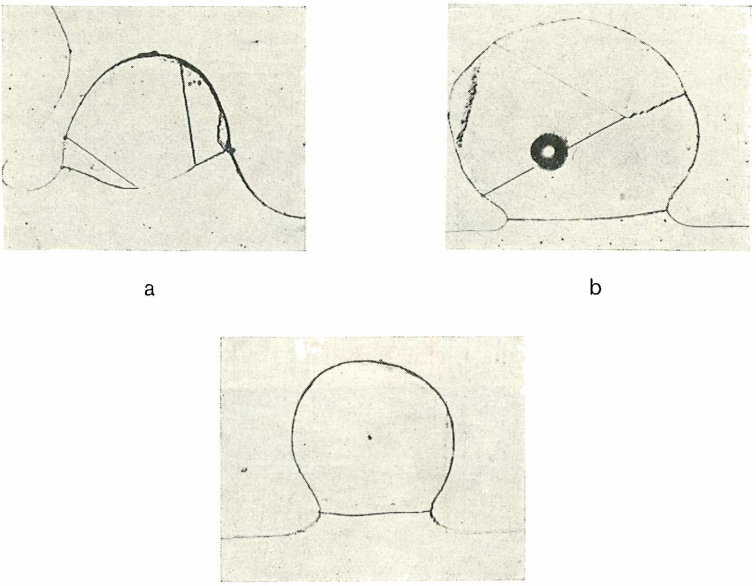
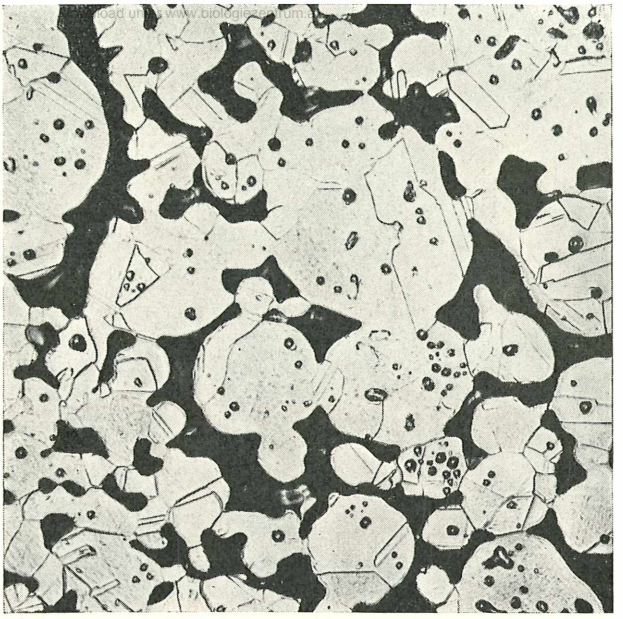


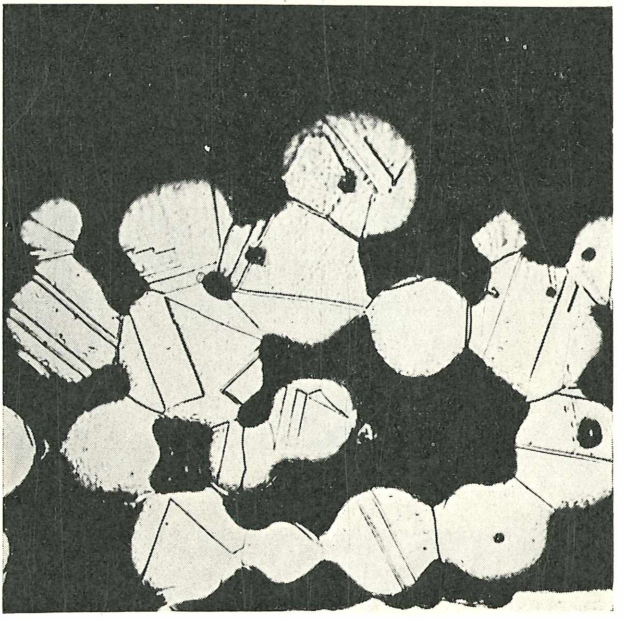

*Fig. 4. Copper Powder on Copper Block.*
*a = sintered at 1040° C. for 16½ hours. Mag. 800:1.*
*b = sintered at 1040° C. for 16½ hours. Mag. 700:1.*
*c = sintered at 1040° C. for 16½ hours. Mag. 700:1.*

of nearly semi-circular profile. The radius of curvature of this profile increases with time and temperature of heating. This radius cannot be measured directly but indirect evaluation is possible as shown in the paper mentioned above (1). As can be seen from the photomicrographs a line connects the middle portions of the semi-circular profiles. This grain boundary represents a surface of minimum cross-section. Sometimes this boundary is curved rather than flat. This is probably due to anisotropy of surface energy at the junction of the particle and the block, which in turn is probably due to the crystallographic orientation at the junction.

*Fig. 5.*
*Copper Powder.*



*a = sintered at 800° C. for 6 hours. Mag. 500:1.*



*b = sintered together at 800° C. for 6 hours. Mag. 500:1.*

Fig. 7, a, b, c, represents the progress of sintering of the globular glass particles at 712° C. for 30 and 40 minutes. The specimens were polished but not etched. Photos were taken with polarized light. Fig. 6 shows the same powder sintered at 760° C. for 5, 10 and 15 minutes. The specimens were etched in a mixture of HF and $HNO_3$. The etching reveals the characteristic difference between the sintering mechanism of the metal and glass particles. Glass particles sintered by viscous flow into the softened glass base and consequently their interfaces are always curved downwards. This sinking is most probably due to the force of gravity and was observed also in the

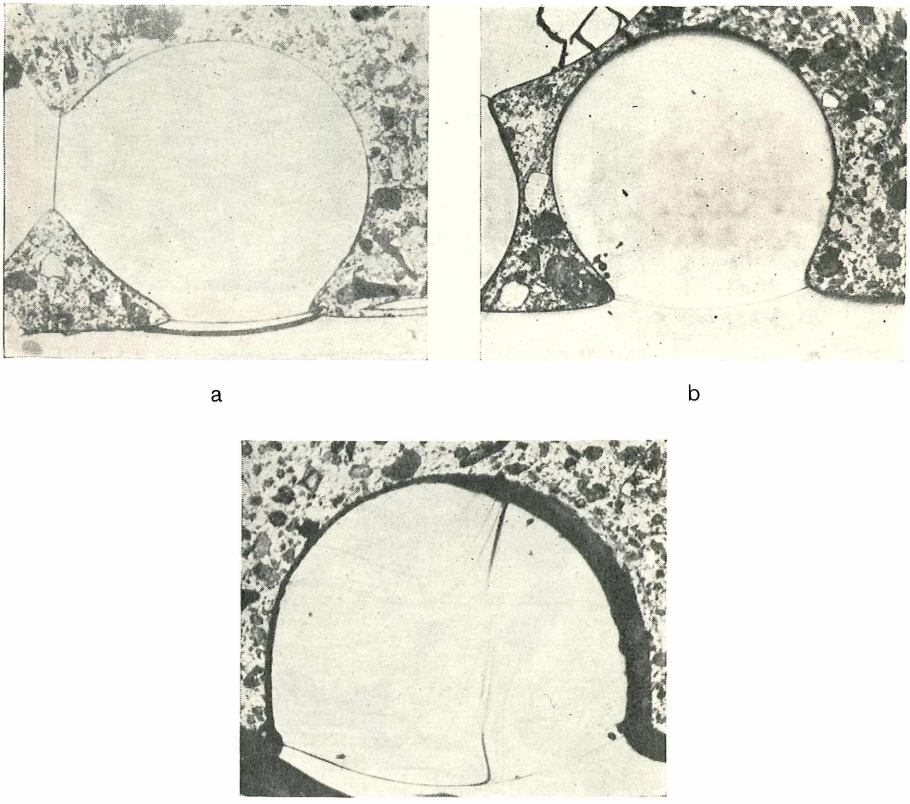
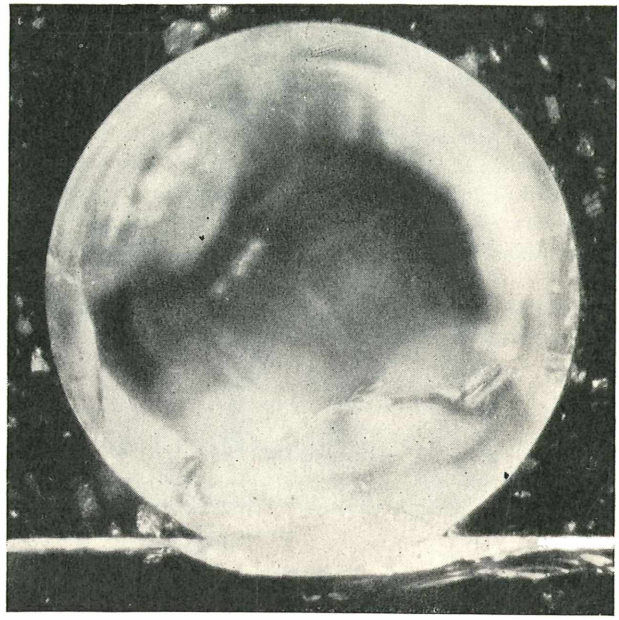
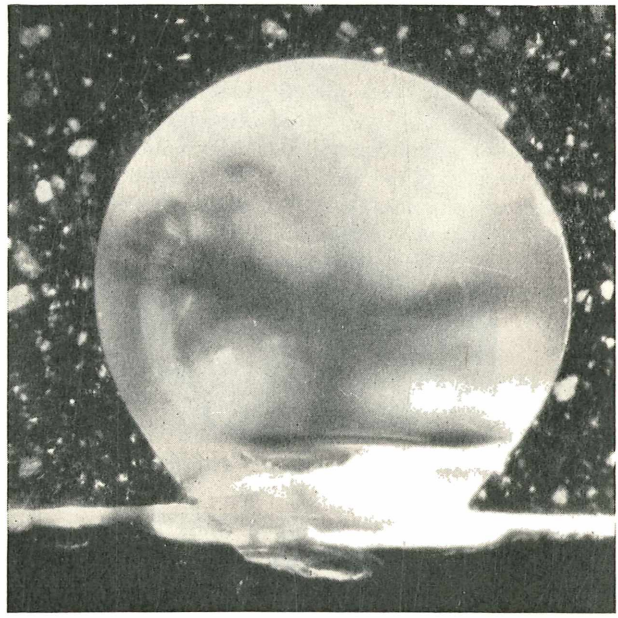



*Fig 6. Glass Powder on Glass Block.*
*a = sintered at 760° C. for 5 minutes. Mag. 250:1*
*b = sintered at 760° C. for 10 minutes. Mag. 150:1.*
*c = sintered at 760° C. for 15 minutes. Mag. 150:1.*

*Fig. 7. Glass Powder on Glass Block.*



*a = sintered at 712° C. for 30 minutes. Photomicrograph taken under Polarized Light. Mag. 150:1.*



*b = sintered at 712° C. for 40 minutes. Photomicrograph taken under Polarized Light. Mag. 150:1.*

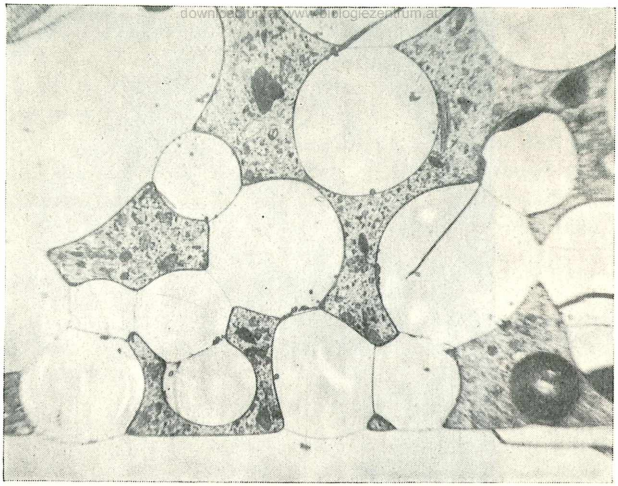


*Fig. 8a.*

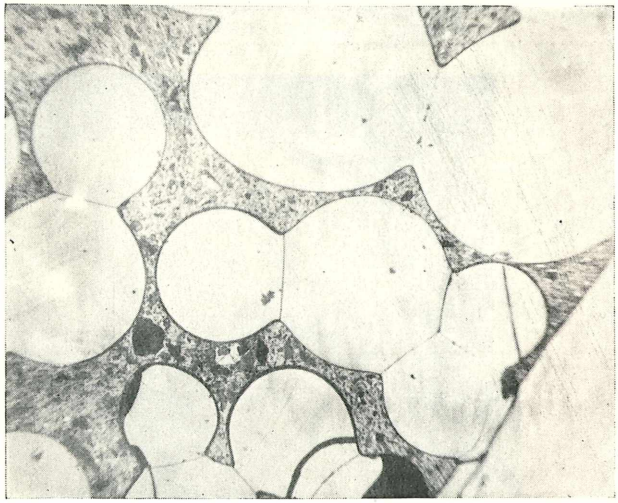


*Fig. 8b.*

*Fig. 8. Glass Powder.*

*a = sintered together at 760° C. for 10 minutes. Mag. 100:1.*
*b = sintered together at 760° C. for 10 minutes. Mag. 100:1.*

case of sintering of wax spheres to a flat wax base. Fig. 8 shows that in the case of mutual sintering of two glass spheres of approximately the same size, the boundary in the absence of force of gravity (direction of sintering is perpendicular to the force of gravity) is straight.

The above described technique was used in a study of sintering of copper, silver and of glass. It was found (1) that the mechanism responsible for sintering of the relatively coarse particles of metals is the volume diffusion. It was also found that for the fine particles and for sintering at relatively low temperature the operating mechanism of sintering is predominantly surface diffusion.

The experiments (2) with glass showed that the viscous flow is responsible, as predicted by FRENKEL (3), for the sintering of particles of this material.

## Acknowledgment

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## Zusammenfassung

Der Vorgang des Sinterns, der in der Pulvermetallurgie eine so bedeutende Rolle spielt, wurde in den letzten Jahren vielfach von Physikern und Metallurgen untersucht. Theorien wurden entwickelt, wonach der Sintervorgang entweder auf Gitterdiffusion, Oberflächendiffusion oder auf viskoses Fließen zurückzuführen ist. Die Verfasser haben den Sintervorgang an Kupfer, Silber und Glaspartikeln studiert, indem sie die Brücken- oder Halsbildung zwischen den einzelnen Teilchen mikroskopisch untersucht haben. Dabei zeigte es sich, daß diese Brückenbildung bei konstanter Sintertemperatur eine Funktion der Zeit ist. Zwischen dem Radius x der zwischen zwei Teilchen gebildeten Brücke und der Sinterdauer t bestehen folgende Beziehungen:

| | |
|---|---|
| Für Oberflächendiffusion | $x^7 \sim t$, |
| für Gitterdiffusion | $x^5 \sim t$, |
| für viskoses Fließen | $x^2 \sim t$. |

Die mikroskopischen Untersuchungen der Verfasser haben an der Halsbildung gezeigt, daß Oberflächen- und Gitterdiffusion die Ursachen des Sintervorganges bei Metallen sind, während viskoses Fließen den Sintervorgang bei Glaspartikeln bedingt.

### Literatur

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3. *Frenkel J.,* Journ. Phys. (USSR) **9** (1945), 5: 385.