Boltzmanns constant and T is the temperature in °Absolute. A measure of the "activation energy" can be obtained from the gradient of a plot of log σ versus $1/T$, as in fig. 1. It is apparent that these measured values of "activation energy" are dependent upon frequency and attempts to use such measurements, obtained from results at any single frequency, for a determination of conductivity mechanisms must be treated with extreme caution.

References

1. F. HUND and R. MEZGER, *Z. phys. Chem.* (*Leipzig*) **201** (1952) 268.
2. E. C. SUBBARAO, P. H. SUTTER, and J. HRIZO, *J. Amer. Ceram. Soc.* **48** (1965) 443.
3. Z. S. VOLCHENKOVA and S. F. PAL'GUEV, *Trans. Inst. Electrochem. USSR* (English Translation) **4** (1964) 152.
4. M. F. LASKER and R. A. RAPP, *Z. phys. Chem.* (*Frankfurt*) **49** (1966) 19.
5. B. C. H. STEELE and C. B. ALCOCK, *Trans. AIME* **233** (1965) 1359.
6. W. E. DANFORTH and J. H. BODINE, *J. Franklin Inst.* **260** (1955) 467.
7. J. RUDOLPH, *Z. Naturforsch* **14a** (1959) 727.
8. C. E. MCGINLEY, Ph.D. Thesis, Glasgow University, 1968.
9. J. E. BAUERLE, *J. Phys. Chem. Solids* **30** (1969) 2657.

Received 30 November 1970
and accepted 9 January 1971

C. E. MCGINLEY*
Department of Metallurgy,
Imperial College, London SW7
P. HANCOCK*
Cranfield Institute of Technology,
Cranfield, Bedford, UK

*Formerly at Glasgow University.

Prolonged Sintering of a High-Permeability Ni-Fe-Cu-Mo Alloy Made by Powder Metallurgy

We have shown recently [1] that the initial permeability of 77 Ni-14Fe-5Cu-4Mo wt% alloy sheet prepared from powder metallurgy compacts can be significantly increased by extending the time of sintering (at 1300°C) considerably beyond that normally used. Conventional sintering times ($\approx$ 5 h) lead to short-range copper concentration gradients in the compact; such gradients are not completely removed by subsequent cold-rolling and annealing, so that the alloy sheet is slightly inhomogeneous. Prolonged sintering leads to a reduction in the degree of inhomogeneity in the alloy compact through the elimination of the concentration gradients.

Previous work [1, 2] investigated sintering times in the range 1 to 156 h. The purpose of this letter is to report some new data which show the effect, on the initial permeability, of sintering for times in the range 100 to 1000 h. The alloy compacts were prepared and heat-treated in a similar way to that described previously [2]. The alloy sheet (nominally 50 μm thick) was annealed at 1050°C for 6 h in pure dry hydrogen, followed by furnace-cooling; the furnace-cooling rate in the important range, 400 to 500°C, was 90°C/h which is the optimum cooling rate for this alloy [3].

The new results are shown in fig. 1. Unexpectedly, the initial permeability *decreases* for

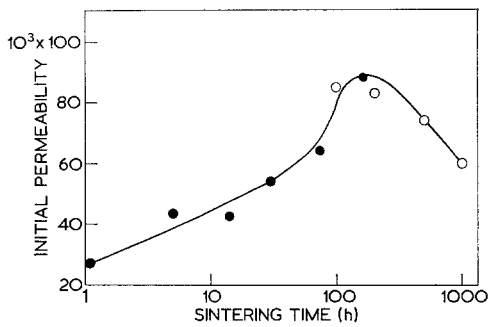

Figure 1 The variation of initial permeability with sintering time for 50 μm alloy sheet cold rolled from sintered compacts and annealed at 1050°C for 6 h. ● previous work [1]; ○ this work.

sintering times greater than 200 h. On the basis of the previous work one of two effects was expected: either (*a*) there would be no change in initial permeability (showing that the concentration gradients had been eliminated) or (*b*) there would be an increase in initial permeability (showing that elimination of concentration gradients was incomplete).

It was subsequently found that prolonged sintering leads to a loss of copper from the compact; for example, after 1000 h sintering the copper content of the alloy sheet was 4.1 wt%. Such a small change in alloy composition would

© *1971 Chapman and Hall Ltd.*

lead to a decrease in the initial permeability in two ways:
(i) Through a change in composition itself [4].
(ii) Through a change in the optimum cooling rate of the alloy; the optimum cooling rate of the alloy sintered for 1000 h was found to be 125° C/h

Electron microprobe analysis showed that evaporation of copper from the compact during prolonged sintering leads to a copper concentration gradient through the thickness of the compact as shown in fig. 2; the compact had rested on an alumina slab during sintering. There is a marked depletion of copper at the surfaces of the compact.

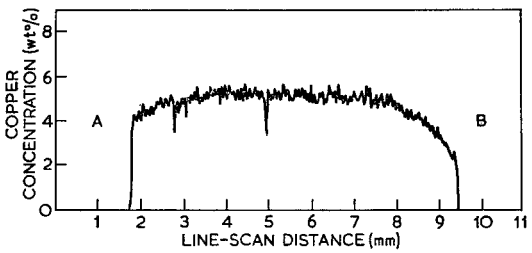

Figure 2 Electron microprobe line-scan analysis showing the variation in copper concentration across a section of a compact (with the composition 77Ni-14Fe-5Cu-4Mo wt.% before sintering) sintered at 1300° C for 1000 h. Side A rested on an alumina slab during the heat treatment.

The presence of this concentration gradient in the compact has an additional effect. It was found that the initial permeability of the sheet obtained from this compact varied along the length of the sheet, being higher at the middle than at the ends. Thus (*a*) the post-sintering "soak" heat-treatment (5 h at 1300° C after cold-rolling the compact to half its original thickness); (*b*) the cold-rolling and intermediate annealing stages down to 50 μm; and (*c*) the final anneal at 1050° C do not completely eliminate the concentration gradient in the compact. Other work [5] shows that, irrespective of the sintering time, the closure of pores by cold-rolling before the soaking stage is particularly ineffective and can be dispensed with, the sintering time being increased accordingly.

Summarising then, three broad regions of sintering times can be identified:
(i) For sintering times in the range 1 to 100 h the inhomogeneity of the compact is caused by the presence of *short-range* concentration gradients associated with copper particles which have melted [2].
(ii) Between 100 and 200 h the compact has low short-range inhomogeneity and negligible long-range inhomogeneity.
(iii) Sintering for times greater than 200 h leads to a loss of copper by evaporation from the surface of the compact, with the subsequent formation of a *long-range* concentration gradient through the thickness of the compact. The effects of the long-range concentration gradient are not completely removed by conventional post-sintering fabrication and heat-treatments.

Acknowledgements

The authors would like to thank Mr A. C. Lynch for helpful suggestions and comments and Mr M. J. Reynolds of Metals Research Ltd who carried out the electron-microprobe analysis. The information in this letter is used by permission of the Senior Director of Development of the Post Office.

References

1. R. D. ENOCH and D. L. MURRELL, *Brit. J. Appl. Phys. (J. Phys. D.)* **2** (1969) 357.
2. D. L. MURRELL and R. D. ENOCH, *J. Mater. Sci.* **5** (1970) 478.
3. R. D. ENOCH and ANNE WINTERBORN, *Brit. J. Appl. Phys.* **18** (1967) 1407.
4. E. V. WALKER, D. K. WORN, and R. E. S. WALTERS, Symp. on Powder Met., Iron and Steel Inst. Special Report **58** (1954) 204.
5. R. D. ENOCH and D. L. MURRELL, unpublished.

Received 5 January
and accepted 15 January 1971

R. D. ENOCH
D. L. MURRELL
Post Office Research Department
Dollis Hill, London, NW2