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QUANTITATIVE SERIAL SECTIONING ANALYSIS OF THE EVOLUTION OF PORE STRUCTURE IN THE LATE STAGES OF SINTERING OF NICKEL POWDER

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ABSTRACT

Quantitative serial sectioning analysis has been applied to the study of the evolution of the microstructure of the pore network during the last ten percent of densification in the sintering of loose stacked nickel powder. This paper focuses upon the combined use of classical stereological counting measurements and topological information obtained from analyzing serial sections. The results demonstrate that the microstructural evolution is surprisingly different from the scenario visualized in conventional wisdom.

Some materials can only be made from powders; others are markedly improved either in performance or economics by following the powder processing route. Applications, particularly in high technology, are increasing. The initial condition in this process is a stack of powder particles. Geometrically this consists of two interpenetrating networks, solid and pore. The process results in the elimination of the porosity. Most products made this way are used in the fully dense condition. The microstructural path traversed is epitomized by the evolution of the pore network and its ultimate disintegration into isolated pores, which then disappear.

The description of this microstructural sequence is geometrically interesting. Ordinary concepts provided by stereology are insufficient for this purpose. Some twenty five years ago, Rhines (1957) presented the basic topological concepts that are suitably adapted to this task: the connectivity of the network (C_V) and its number of disconnected parts (N_V). Stereological estimation of these topological properties has been shown to require serial sectioning analysis. (Aigeltinger and DeHoff (1975)).

From a topological point of view, the most interesting part of the process is the late stage wherein the connected network of porosity breaks up into isolated pores. There is evidence that the path by which this is accomplished significantly affects the attainment of full density: thus, this stage of the process is of practical importance. The present investigation focussed upon nickel samples with porosities in the range from 10% toward 2%, prepared by sintering loose stacks of a nominally 5 micron equiaxed nickel powder for various times in dry hydrogen at 1250°C. Standard stereological procedures were applied to estimate:

V_V, the volume fraction of porosity

S_V^{PS}, the area of pore solid interface

S_V^{SS}, the area of grain boundaries

M_V^{SP}, the integral mean curvature of pore-solid surface

L_V^{SSP}, the length of solid-solid-pore triple lines

L_V^{SSS}, the length of grain edges

f_V, f_S, f_1, fractional occupancies of grains, grain boundaries and grain edges by porosity

The connectivity and number of separate pores were measured on each sample by serial sectioning.

The serial sectioning analysis provided a unique opportunity for a new level of analysis of the structure. During the network disintegration stage, regions of the porosity become isolated from the network as their connecting channels pinch off. By selecting a micrograph from the middle of the stack of serial sections, it was possible to assign the pore sections observed thereon to two categories.

a. Those that are part of the remaining network, and
b. Those that are part of the collection of isolated pores.

Once this discrimination is established on a section, the standard stereological measurements could be used to estimate V_V, S_V and M_V separately for "network character pores" and "isolated pores." The results were a surprise, see Figure 1.

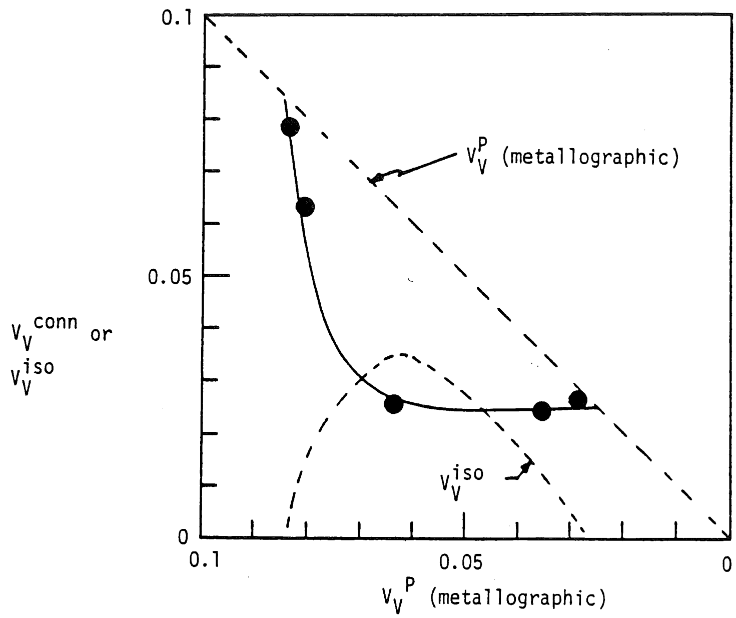

Figure 1. Variation of the volume fraction of total network and isolated porosity in the late stages of loose stack sintering of nickel powder.

Conventional wisdom in powder processing had concluded that network porosity will continue to evolve during the late stages of the process. In contrast, pores that had become isolated are expected to be very difficult to eliminate. Figure 1 shows that at $V_V = 0.08$ virtually all of the porosity remains connected in the network. At $V_V = 0.06$, two-thirds of the pore volume has been isolated [0.04]; one-third [0.02] remains as a connected network. This change reflects the expected disintegration of the network. However, at a later state when only 0.02 volume fraction of porosity remains, <u>all of it is network porosity</u>. This is the same quantity of network porosity [0.02] that had remained in the sample containing a total $V_V = 0.06$. The porosity that disappeared in the evolution between the two samples at 0.06 and 0.02 total volume fraction <u>was exclusively the isolated porosity</u> that existed at $V_V = 0.06$. Values of S_V and M_V for the two classes of pores showed similar behavior.

The explanation of this contradiction with existing conventional wisdom has pointed the way to a new level of understanding of this important stage of powder processing. However, that is not the point of this presentation. From a stereological point of view, this study exhibits a strategy that combines serial sectioning analysis, which provides a topological basis for discriminating the structural features, with standard stereological procedures for assessing metric aspects of the discriminated parts. The result provides a level of understanding not otherwise attainable.

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REFERENCES

Aigeltinger E H and DeHoff R T. Quantitative Determination of Topological and Metric Properties During Sintering of Copper, Met Trans. GA (1975) 1853.

Rhines F N. A Topological Model of Sintering , in Plansee Proceedings, 3rd Seminar, Reuttle, Tyrol (1958) 38.