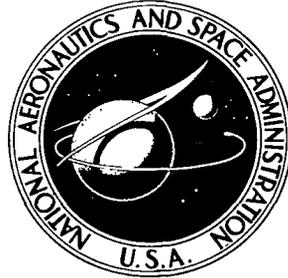


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METAL STRUCTURES WITH PARALLEL PORES OF UNIFORM SIZE

by J. Sherfey

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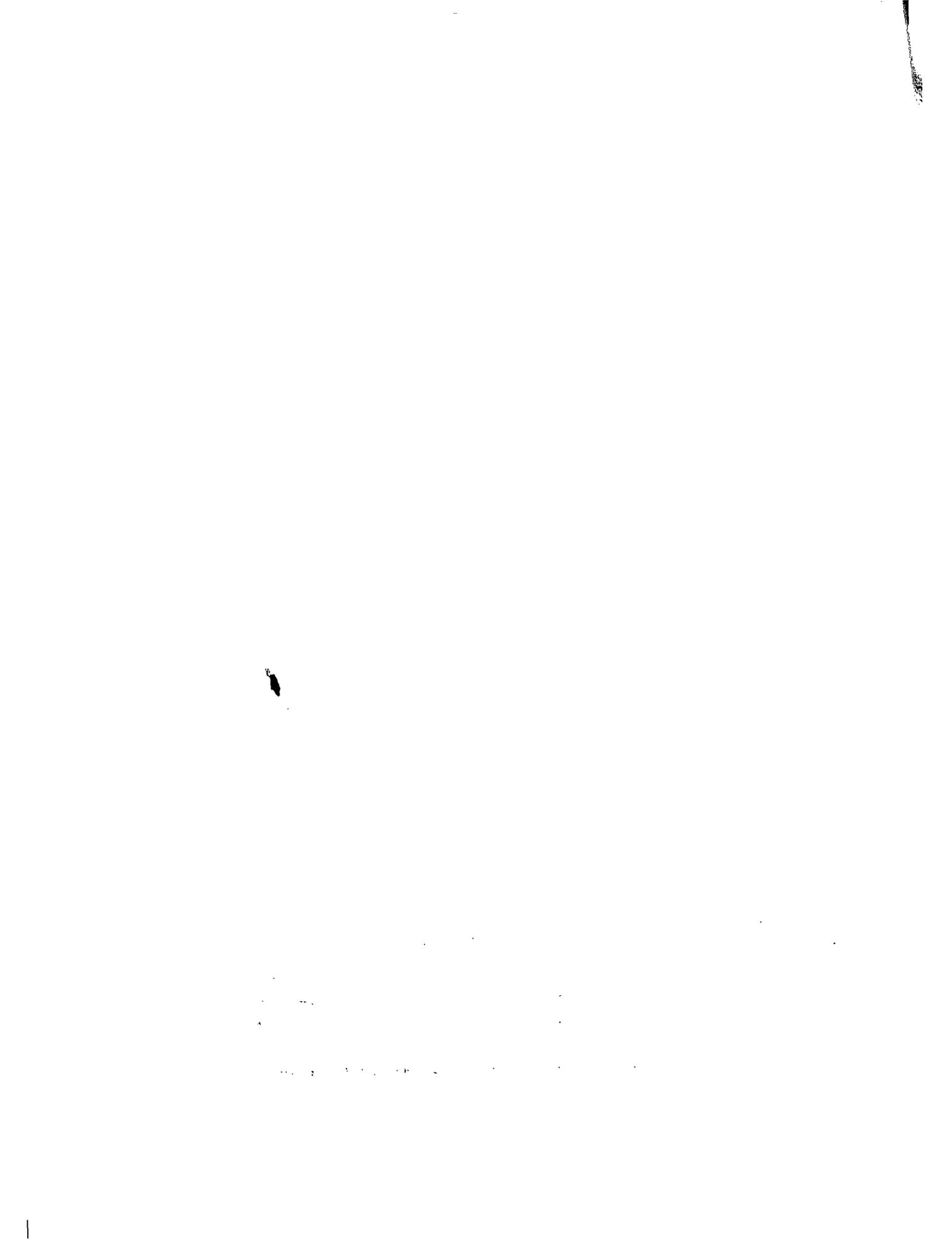


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16. Abstract A wide variety of techniques for the preparation of metallic structures with parallel, uniformly sized pores is described critically. One of these techniques is already employed commercially, but others may be totally impractical. The method chosen will depend on the characteristics of the structure to be fabricated. Some of the processes described are applicable to materials other than metals.			
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METAL STRUCTURES WITH PARALLEL PORES OF UNIFORM SIZE

by

J. Sherfey

Goddard Space Flight Center

INTRODUCTION

The fabrication of porous metal parts by the techniques of powder metallurgy is an old and well-established industry. It is only recently, however, that methods have been developed which make it possible to prepare such structures with parallel pores of uniform size. One company* has made these parallel-pore materials available on a semi-commercial basis for experimental and evaluation purposes. Even though there is presently no large-volume commercial use for such structures, there is widespread interest in them, and numerous potential applications have been proposed. Indeed, these materials have been described as "a solution in search of a problem".

The field of electrochemistry affords two potential applications, the plaques of sintered plate cells and the electrodes of certain types of fuel cells. These devices are now fabricated by means of conventional powder metallurgical techniques, and are, therefore, characterized by a random pore structure. In both of these examples, the electrochemical reactions involved take place largely within the pores of the structure. Also, in both cases the detailed mechanisms of the chemical and physical processes which occur are imperfectly understood. Nonetheless, it seems logical to assume that there is an optimum pore geometry for any given application and that this optimum is not attained by the structures now being used. This line of reasoning can be extended to include the hypothesis that the pores in such a device, if optimized, would be uniform in size and perpendicular to the plane of the electrode. Certainly, such structures would be most useful for theoretical studies of porous electrodes. Several workers have, in fact, reported researches based on this idea (References 1 and 2).

Considerations such as these prompted the author to explore the possibility of fabricating such structures with uniformly sized parallel pores. This paper is a report on that effort. It reviews and evaluates the techniques currently available; it describes several new approaches which are now in the process of development; and it proposes several new fabrication methods which appear to have promise.

*Brunswick Corporation, Corporate Research and Development, Needham Laboratories, Needham, Mass.

The electrochemical applications mentioned previously place certain limitations on the properties of the structures considered here. For example, the electrodes desired are planar or sheet-like objects with perpendicular pores and, ordinarily, a rectangular outline. The thickness can vary, depending on the application, from a few hundredths of an inch up to as much as a quarter of an inch. A relatively high degree of porosity is ordinarily required, perhaps 50 to 90 percent. The optimum pore diameter for some applications might be as large as 100 μm (4×10^{-3} in.) and for others as small as 0.1 μm (4×10^{-6} in.). Nickel is the preferred material of construction for most of the uses contemplated, but the techniques described are applicable to a broad spectrum of metals, alloys, and other materials.

The various methods of fabrication to be discussed fall conveniently into five categories, including one catchall or miscellaneous group. The other four have been assigned essentially trivial names such as "Elongate-Bundle" for ease of reference.

FABRICATION TECHNIQUES

The Elongate-Bundle Approach

The first approach to be considered, called the Elongate-Bundle approach, is the only one that has been reduced to commercial practice. At least two companies* are using this technique to fabricate metal structures with uniform parallel pores. The details of the process have not been published, but the basic idea can be grasped by means of the following example.

The starting material is a copper rod that has been clad with a thick-walled nickel tube. The latter is then machined to obtain a billet having a square cross section. This billet is elongated by a factor of 81, cut into 81 equal lengths, and then reassembled into a 9 by 9 array to form a new billet approximating the original in size but containing 81 copper rods, each 1/9 the starting diameter. This process can be repeated one or more times to obtain the necessary reduction in the size of the copper. The billet is then cut into slices perpendicular to the axes of the imbedded copper rods, and the desired structures are obtained by leaching the copper out of these slices. The minimum pore size that can be obtained by this technique is limited by the interdiffusion of the two metals and by the ability of the leaching solution to penetrate a long thin pore. It has been shown* that it is possible to prepare stainless steel structures with pores that are approximately 4 μm (0.00016 in.) in diameter and 100 μm (0.004 in.) long.

This is a flexible process that can be used to produce a variety of structures. For example, this technique can be made to yield a structure like that shown in Figure 1 by introducing two modifications. First, the copper and nickel are interchanged in the starting material, and second, the slices are sintered to a backing sheet before the copper is removed. Without such a backing sheet, the structure would, of course, fall apart.

*Brunswick Corporation, Needham Laboratories, Needham, Mass., and Whittaker Corporation, Nuclear Metals Division, West Concord, Mass.

The Wind-and-Sinter Approach

Another method, which can be called the Wind-and-Sinter approach, can be exemplified by the following process. Fine copper wire is coated with a nickel electrodeposit and then is wound onto a spool and sintered to form a coherent mass. After the winding is removed from the spool, the wire coil is cut perpendicular to the wires to yield slices of the desired thickness. Finally, the copper is leached out, leaving a structure having the appearance of Figure 2.

In its details, this basic procedure is subject to numerous variations and improvements. It can be shown, for example, that the electrodeposition process becomes impractical for fine wires if one considers both the length of wire involved per unit weight of product and the current-carrying capacity of the wire. This difficulty can be circumvented by electroplating a relatively heavy wire and drawing the composite. Even better, one could start by cladding a copper rod with a nickel tube and drawing this structure to the required diameter. Nickel-coated copper wire made by the latter process is available commercially.*

In some applications a modification of the above process might offer advantages. Instead of starting with nickel-coated copper wire, a three-layer structure consisting of an inner copper core, a nickel overlay, and a second overlay of copper could be used. This could be wound, sintered, and sliced as before. The slices are then sintered or brazed to a nickel backing sheet. As a final step, the copper is leached out, leaving a structure with the configuration illustrated in Figure 3. Of course, it would be possible to cover both sides of the backing sheet instead of only one. The basic idea behind this modification is not unlike the one suggested under the Elongate-Bundle approach.

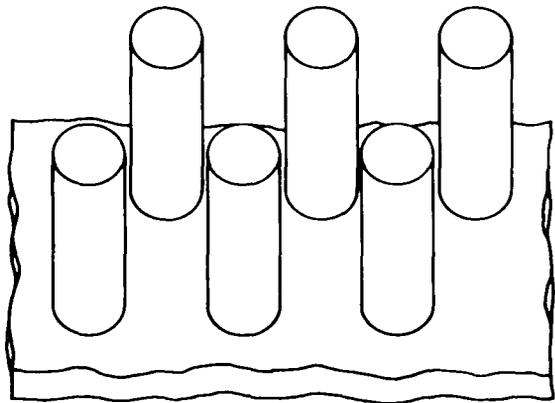


Figure 1—One possible structure produced by the Elongate-Bundle approach.

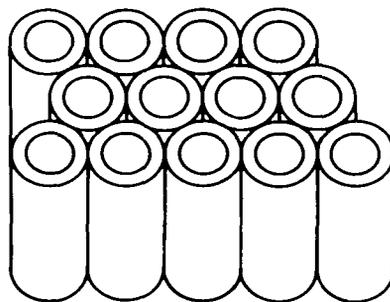


Figure 2—The structure produced by the electrodeposition method of the Wind-and-Sinter approach.

*General Telephone and Electronics, Sylvania Parts Division, Warren, Pa.

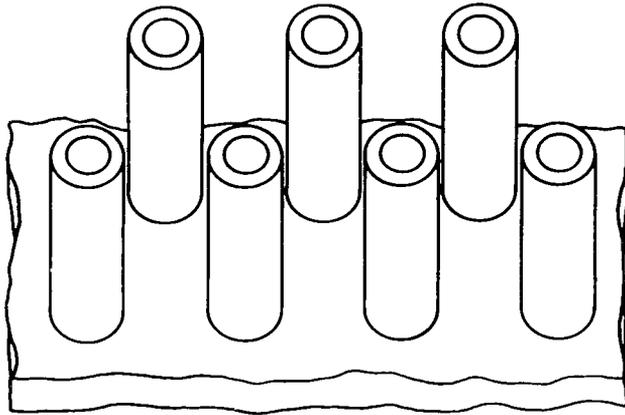


Figure 3—Another possible structure that could be produced by the Wind-and-Sinter approach.

W. Kuhn has patented a process for fabricating small-diameter metal capillaries.* This product could act as a starting material for the Wind-and-Sinter approach. The process Kuhn describes is quite flexible but can be explained by the following specific example. A slurry containing a metal powder and a volatile liquid is used to coat a filament made of an organic polymer. The coated filament is then heated in such a way as to decompose and volatilize the filament while sintering the powder into a porous tube. This patent also covers the use of carbon or glass as a filamentary material. The former could be burned away and the latter could be

removed by leaching. If used to prepare a nickel structure with parallel pores, a glass or silica filament would be the preferred starting material because either one of these materials could be removed in a final step by leaching with hydrofluoric acid. Glass and silica are both soluble in this reagent, but nickel is not attacked.

For some applications, this process could be modified advantageously by the incorporation of a small quantity of a brazing material with the powdered metal. This technique would make it possible to effect a large reduction in furnace residence time because the tube-forming process would involve melting, which can be very rapid, rather than sintering, which is inherently slow. The braze could be removed at a later stage in the processing if a porous capillary wall is desired. For example, if cadmium were used as a braze, it could be evaporated. Other brazing materials could possibly be leached.

The sintering step in the Wind-and-Sinter approach can be replaced with other techniques for consolidating the winding. Recent interest in fiber-metal composites has led to the investigation of a variety of methods for depositing a metal on a spool or mandrel as it is wound with a filament (Reference 3). Withers and Abrams (Reference 4), for example, have demonstrated the feasibility of an electroforming process. They wound the mandrel with a filament while it acted as the cathode in a metal plating bath. By suitable adjustment of such variables as the rates of winding and electrodeposition, they were able to produce void-free composite structures.

There are several weaknesses in the Wind-and-Sinter approach. One serious limitation is the cost of fine wire. One alloy investigated, for example, was priced as shown in Table 1.

Materials such as fused silica, glass, carbon, and various synthetic textile fibers are available in sizes down to about 10 μm (0.0004 in.) and are comparatively inexpensive.

*Kuhn, William E., "Foraminous Product and Method for Making Same," U.S. Patent Application S.N. 775,471.

Table 1—Sample alloy wire pieces.*

Diameter (in.)	Cost per lb
0.010	\$12.36
0.004	\$19.11
0.001	\$262.00
0.0005	\$1,176.00
0.0004	\$4,500.00

It can be seen that the cost factor greatly increases the attractiveness of processes such as slurry coating and electroforming because these can both be adapted to the use of less expensive materials.

The product obtained from the Wind-and-Sinter approach will not ordinarily conform to the geometrically perfect array illustrated in Figure 2. Such a pattern would result only if the coil winding machine were to perform flawlessly, and, to quote a manufacturer of these devices, "...perfect layer winding is oftentimes an extremely expensive requirement to meet, and one which, under certain circumstances, is virtually unobtainable, regardless of cost."**

As a final objection, it should be noted that axial cuts in a helical coil yield wedge-shaped slices. Slices with parallel sides can be cut, but the process necessarily involves a considerable amount of waste. Elliptical or rectangular spools would probably help, but there seems to be no way to completely circumvent this problem without building a device that would cut and stack straight lengths of fibers. The design of such a device would not be simple.

The Extrude-and-Sinter Approach

The essential aspects of the Extrude-and-Sinter technique can be seen in the following example. A heavy slurry or paste is prepared from water, chopped fused quartz fibers, nickel metal powder, a wetting agent, and a thickener or plasticizer. This is forced through a small orifice, thus aligning the quartz fibers into a parallel configuration. The extruded material is cut into convenient lengths, stacked parallel in a suitable container, and compacted while wet to remove entrapped air. The wet compact is fired first at a low temperature to remove water and organic matter and then at some higher temperature to sinter the nickel powder.

*Diameter is given in inches rather than micrometers, and cost per pound is shown rather than cost per kilogram, because these are the units used by the vendor.

**Howard A. George, Coil Winding Equipment Co., Oyster Bay, N.Y. (private communication).

This technique for aligning fibers was first described by L. Frank and H. Barr* (Reference 5), who used it to prepare high-strength composites that were devoid of pores. For the present areas of application, however, complete compaction is neither necessary nor desirable. Instead, the billet is partially compacted, sintered to increase strength, and then cut into slices with the quartz fibers perpendicular to the plane of the slice. Figure 4 is a photomicrograph of the surface of such a slice. The cut ends of the 10 μm (1/2 mil) quartz fibers appear as black disks. The lighter background is sintered nickel powder having a porosity of about 30 percent. The desired structure with parallel pores is obtained by dissolving the quartz in hydrofluoric acid.

The feasibility of this approach has been demonstrated in an investigation which is currently in progress. ** The details of this project will be reported separately.

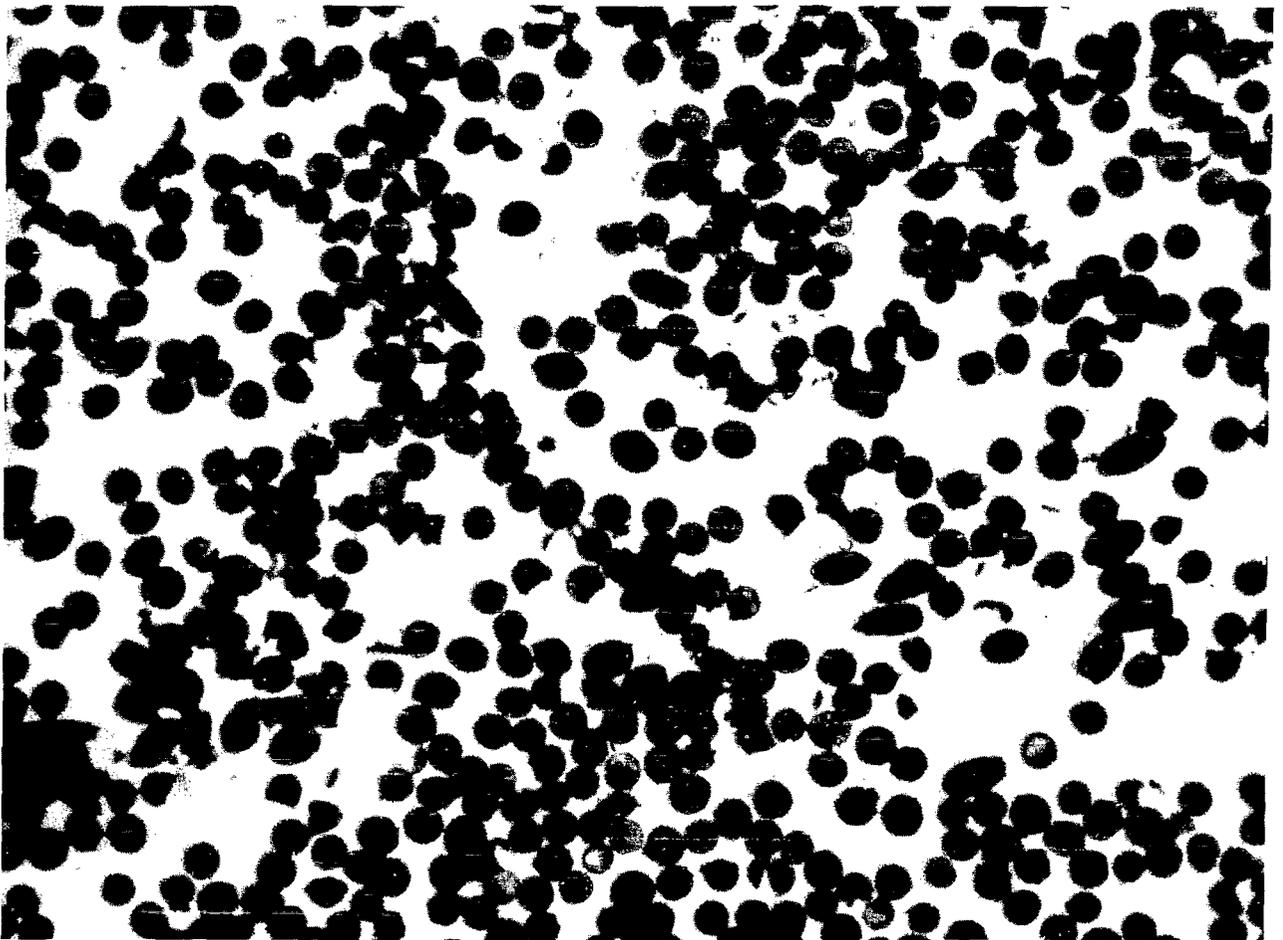


Figure 4—Photomicrograph of the surface of a cross sectional slice of a billet prepared by the Extrude-and-Sinter approach.

*Barr, H., and Frank, L., "Method for Making Oriented Fiber on Whisker Composites," U.S. Patent No. 3,432,295, March 11, 1969.

**NASA Contract No. NAS-5-9479, with Hittman Associates, Inc., Columbia, Md.

It should be noted that this fabrication process yields a structure having two kinds of pores: induced pores caused by the removal of the quartz filaments, and interstitial pores resulting from the incomplete compaction of the powder-metal matrix. Also, the amount of porosity from each source can be controlled and the diameter of the induced pores is essentially uniform. This dual porosity is also characteristic of the slurry coating variations of the Wind-and-Sinter approach previously described.

The details of the fabrication process and the nature of the materials used in the Extrude-and-Sinter approach can both be varied to meet the needs of a specific end product. The basic process could be used, for example, to produce sieves having holes smaller than any practical sieve currently available. Here, it would be necessary to have essentially straight induced pores with smooth walls. Otherwise, clogging of these pores would almost certainly be a problem. Such straight, smooth, induced pores could be obtained by avoiding mechanical compaction and, instead, filling the interstitial pores with a braze prior to removal of the pore-former.

In some applications it would be desirable to reduce the diameter of the induced pores to a micrometer or less. The fuel cell electrode is perhaps a case in point. All of the potential pore-formers investigated (glass, quartz, metal wire, and the various synthetic organic fibers) are available in diameters as small as 5 to 10 μm , but it seems that not one of these materials is available in a size range less than this.

This dilemma could perhaps be resolved by combining the Elongate-Bundle and the Extrude-and-Sinter approaches. Thus, one could start with 100 μm (0.004 in.) copper wire as a pore-former. This material is comparatively inexpensive and easy to handle. Using the Extrude-and-Sinter approach, this wire could be dispersed as short parallel segments in, for example, a nickel billet. The Elongate-Bundle approach could then be used to effect two consecutive 10-fold reductions in the billet cross section, thus reducing the diameter of the copper wires from 100 to 1 μm . The final steps would involve slicing and leaching as previously outlined.

A number of interesting possibilities are raised if one introduces the idea of coating the pore-former (wire, glass filament, etc.) before using it in the Extrude-and-Sinter approach. For example, copper wire coated with a platinum electrodeposit could be used to fabricate a nickel fuel cell electrode with platinum-lined pores. This coating technique would be beneficial if the end product were to be a sieve. Thus, if silica fibers were coated with nickel metal before this composite is used to prepare a nickel sieve, one could be assured that the largest hole diameter would be the same as the largest quartz filament, because the clumping of two or more filaments would not cause the formation of an oversize pore.

Finally, it should be noted that the Extrude-and-Sinter approach is applicable to materials other than metals. Ceramics and plastics are likely candidates.

The Corrugate-Stack Approach

The Corrugate-Stack approach was first proposed by J. McCallum et al. (Reference 1) as a potential technique for the fabrication of plaques for the nickel cadmium cell. Like the Extrude-and-Sinter approach, it is the basis for a current investigation, the details of which will be reported separately. * This paper will discuss only the basic idea and a few of the more significant findings.

In its essence, the procedure is as follows: Corrugated metal sheets are electroformed and then stacked with the grooves of all the sheets running parallel. This assembly is sintered to form a coherent mass and then sliced in a direction such that the plane of the slice is perpendicular to the grooves formed by the corrugations. The slices thus obtained are the desired parallel-pore structures.

Present indications suggest that this is a thoroughly practical process, but it is not without its difficulties and limitations. One problem which is immediately apparent is the fabrication of sheets with both a metal thickness and a corrugation depth of the order of $25\ \mu\text{m}$ (10^{-3} in.). This has been solved by means of an electroforming technique, but the handling of this material in the absence of special machinery is a tedious hand process. It can be seen that the need for a high degree of porosity, e. g., 70 to 80 percent, constrains any effort to minimize this difficulty. Present indications are that the Corrugate-Stack approach is not a practical route to pore sizes less than $25\ \mu\text{m}$ (0.001 in.).

The second difficulty is the tendency of adjacent sheets to nest, thus obliterating the pores. One seemingly effective solution to this problem is offered in Figure 5, which illustrates that the use of alternate layers of uncorrugated sheet prevents such nesting. Also, note that the corrugations in any given sheet make an angle of about 30 degrees with those in the corrugated sheets immediately above and below. This angling of the corrugations with respect to those above and below increases the compressive strength of the assembly. With parallel corrugations, these structures tend to be distorted when subjected to a compressive stress during sintering, as shown in Figure 6.

Experience with slicing this type of material is limited, but it seems probable that it will be necessary to fill the voids with wax or some similar substance before cutting. This could be removed subsequently by melting and/or solvent extraction.

Figure 7 is a photomicrograph of a finished structure. The peak-to-peak distance of the corrugations in this structure is $250\ \mu\text{m}$ (0.01 in.).

Miscellaneous Approaches

This category of fabrication techniques includes all those approaches which seem to have less merit in present applications than those previously described. They are included here for the sake of completeness and because these methods might take on a greatly enhanced attractiveness to the reader with a different end-use in mind.

*NASA Contract No. NAS-5-21105, with Battelle Memorial Institute, Columbus, Ohio (Final report to be published).

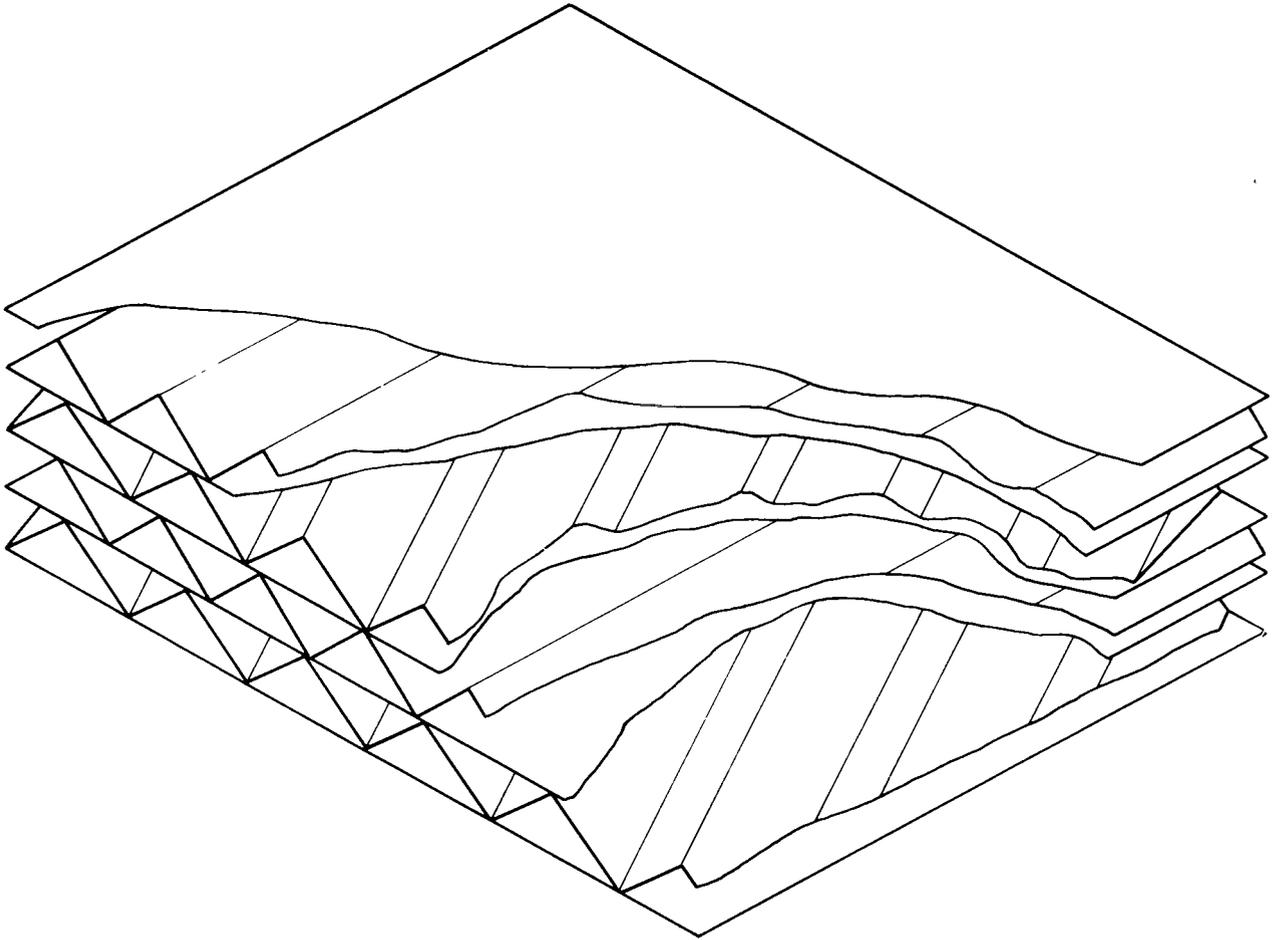


Figure 5—The deployment of non-parallel corrugated layers to prevent nesting in the Corrugate-Stack approach.

The Stacked-Screens Approach.

McCallum et al. (Reference 1) have investigated a fabrication technique based on the use of stacked electroformed screens. The fine-mesh screens required are available commercially* or they can be prepared by conventional electroforming or photoengraving techniques. The interested reader is referred to the references just cited or to the manufactures of the photoresist materials that are commonly used in an essential step in these processes.

These techniques for preparing screens cannot be modified to yield porous bodies of the type under consideration here, i. e. , relatively thick structures with long, thin pores. McCallum circumvented this problem by superimposing one screen on another until the

*Buckbee-Mears Co., St. Paul, Minn.

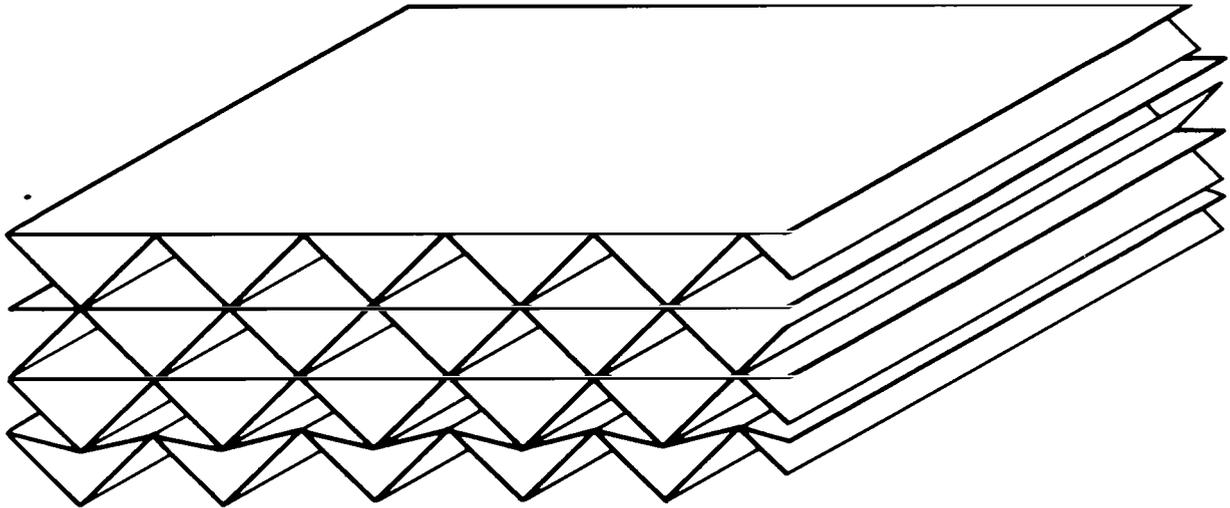


Figure 6—Distortion caused by compressive stress on parallel corrugations in the Corrugate-Stack method.

required thickness, or pore length, was attained. This was done while alignment or registration of all the layers involved was maintained. In this way, an assembly with straight pores was obtained. The apparatus developed to accomplish this was mounted on the stage of the microscope used to observe the position of each succeeding layer. After being so assembled, the mass was sintered to form a coherent body.

It is obvious that this technique, even though it is both ingenious and effective, is not suited to the production of parallel-pore structures in any quantity greater than that needed for small-scale laboratory experiments.

The Flock Approach.

The word "flock" is used to describe a process in which short lengths of textile fibers are dropped onto an adhesive-coated surface to obtain a matte or felt-like finish. In a refinement of this technique, the fibers are dropped through an electrostatic field, thus aligning them perpendicular to the surface. The velvet finish usually applied to jewelry boxes, for example, is obtained in this way.

This process can be conceived as being duplicated with metal in place of fabric, thus producing a structure like that shown in Figure 1. The feasibility of this technique was explored in a rather crude series of experiments in which short lengths of fine nickel wire were dropped through a magnetic field generated by a solenoid. The results of these experiments were inconclusive, but at least one problem was encountered, a tendency of the "fibers" to clump together, and other problems were anticipated. None of these difficulties seem to be insurmountable, but the flocking of metals must nonetheless be viewed at the present time as an interesting but unproven technique.

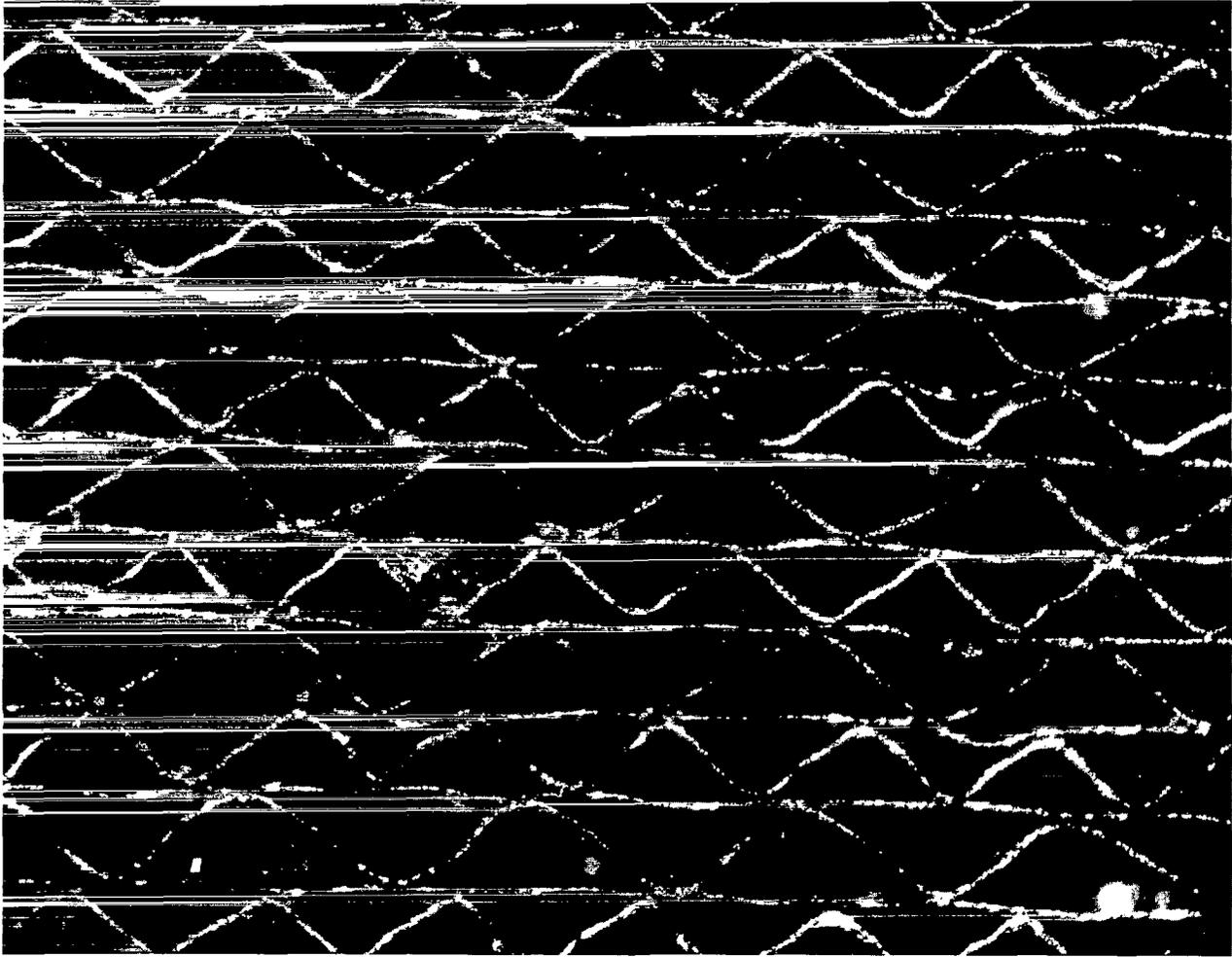


Figure 7—Photomicrograph of a finished structure produced by the Corrugate-Stack approach.

The Weaving Approach.

Metal wire cloth is commercially available in a wide variety of weave patterns, metals, and wire sizes. It is obvious that, by stacking and sintering, some of these could be used to prepare structures which would be described as having parallel pores. A more sophisticated, but similar, approach would replace the conventional loom with a computer-controlled filament-winding machine of the type that has been developed to form fiber-reinforced composites. Any such weaving process, if used to prepare structures of the type contemplated here, is subject to criticism on at least two counts, the cost of the wire and the enormous lengths that would be required per unit weight of product. A pound of 2-mil nickel wire, for example, is 60 miles long. The relative weakness of such fine wire might also present a formidable problem. It is clear that the various weaving processes are not very practical when viewed in the light of present requirements.

The Metallized-Velvet Approach.

Pile-type fabrics, such as velvet and the oriented flock-type material just mentioned, are available in great variety and are relatively inexpensive. Bauman* has suggested that these materials could be metallized to obtain a parallel-pore structure by using the parallel fibers of the nap as a pore-former. The practicality of this general approach has been demonstrated by Botosan and Katan (Reference 6), who metallized a felt-type fabric and then tested it as a fuel cell electrode. Justi** has investigated a somewhat similar method, which also uses a pile-type fabric as a pore-former. He fills the voids in the fabric with metal powder, which is subsequently consolidated by mechanical compaction or sintering. The fabric skeleton can be either burned off or left in place, depending on both the metal involved and the particular application.

This technique has several obvious limitations and is certainly less versatile than others discussed above. It would perhaps be attractive in some special applications.

The Alginate-Honeycomb Approach.

H. Thiele and A. Wiechen have described an interesting process based on the replication of alginate film (Reference 7). By means of appropriate chemical processing†, such films can be prepared with pores that are normal to the film surface, essentially identical in diameter, and with a remarkably uniform distribution over the face of the film. It is claimed that pore size and degree of porosity can both be controlled and varied over a wide range.

After they have been strengthened by a rather complex process involving both dehydration and plastic impregnation, these films can be metallized to obtain a similar structure in, for example, nickel. The organic skeleton can then be removed, if necessary, by a process such as pyrolysis or leaching.

It is difficult to evaluate the practicality of this approach without first-hand experience with the processes involved. The references cited indicate that the steps are numerous but easily controlled and subject to automation. Certainly, this approach offers at least one significant advantage over most of the approaches previously described, in that there is no slicing step. If the structure required is relatively thin but large in area, this step can be very wasteful and difficult or even totally impractical. The Alginate-Honeycomb approach obviates this limitation and places no practical restriction on the area. In short, this is an attractive approach which should be evaluated by anyone interested in parallel-pore structures.

Goddard Space Flight Center
National Aeronautics and Space Administration
Greenbelt, Maryland, November 9, 1970
120-34-11-06-5 1

*H. Bauman, Lockheed Missiles and Space Company, Materials Sciences Laboratory, Palo Alto, Calif. (private communication).

**Justi, F., "Homoporous Gas-Diffusion Electrode for Galvanic Cells," U.S. Patent No. 2,860,175, November 11, 1958.

†Thiele, H., "Membranes and Shaped Articles Prepared from Polyelectrolytes," British Patent No. 918,626, Feb. 1963.

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