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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This report summarizes efforts and accomplishments in the areas noted in the title. Structural analysis of Liquid Phase Sintering materials is clarified in terms of solid phase contiguity and scale; the latter being predictively so. Mechanisms for structural evolution in LPS products are also explained. Mechanical processing capabilities of in situ metal matrix composites are developed and the properties obtained in them are measured and compared with theories for the same. Guidelines for developing high strength cold worked metal matrix composites are presented.

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Final Report

Microstructural Development and Control in Liquid
Phase Sintering; Processing, Structures and
Properties of Cold Worked Metal Matrix Composites

T. H. Courtney
J. K. Lee

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I. INTRODUCTION

Research effort on this program has focused on two separate, but related, areas: 1) microstructural development and control in liquid phase sintering (LPS), and 2) processing, structure and properties of heavily cold worked in situ metal matrix composites. Microstructural studies on LPS can be divided further into the areas of densification, microstructural morphology and microstructural scale. In this program, the latter two facets of LPS structures were studied most extensively, although some attention has also been paid to the important area of densification, particularly the last stage of it. The metal matrix composite research developed originally as a derivative study from our work on LPS materials. We wished to illustrated that LPS is an appropriate processing vehicle for producing initial structures capable for fabrication into metal matrix composites. These efforts rapidly developed into a program with considerable importance in its own right. In fact, it has occupied almost the whole of our efforts in the past twelve to eighteen months and has, we believe, led to further understanding in processing capabilities of such materials, the development of criteria for selection of materials suitable for composite fabrication, and approximate predictive capabilities for the strengths obtained in this class of composites.

This report summarizes briefly the major accomplishments we believe we have made in these two research areas. Inasmuch as most of the work involving LPS has been reported already in the open literature, only the most significant results will be described here. On the other hand, the findings of the studies on metal matrix composites are just now beginning to be submitted to, and appear in, journals. Therefore, the results from this work will be presented here in somewhat more detail.

II. Summary of Results

A. Microstructural Development and Control in Liquid Phase Sintering

As noted three parameters -- density, morphology and microstructural scale -- define the structure, and hence properties, of LPS materials. In turn two features -- solid particle shape and solid phase contiguity -- define the morphology of a LPS alloy. Nonspherical, or faceted, particles are present when the solid-liquid interfacial energy is anisotropic. Alternatively, spherical particles may become "flattened" as a result of space filling considerations when the solid phase volume fraction (V_p) is large, such as it is in many of the heavy metal alloys. We have found (1) that solid phase contiguity is not system specific if it is measured in terms of the number of solid-solid particle contacts per particle (C_p). That is C_p is a function only of V_p and, moreover, is unaffected by sintering time or temperature, provided time is long and temperature high enough so that C_p is evaluated beyond the first stage of sintering. On the other hand the average contact area (usually normalized in terms of the particle surface area) is system specific and when contiguity is measured in this way, it is likewise so.

Both results mentioned above are of significance. That C_p is fairly high (a skeletal structure forms when $V_p \geq 0.20$) and system independent is evidence that a large number of solid-solid contacts are made during the sintering process, and that a certain (small) fraction of these "stick", this in spite of the requirement of solid phase wetting necessary for densification in LPS materials. Small differences in density between the solid and liquid phases produce this great frequency of solid particle collisions and that but a small number of them result in adherence between particles is a manifestation of the tendency for the liquid to wet the solid. This wetting condition results also in boundaries formed thusly being of the low energy and small misorientation

type (special boundaries). This feature of LPS had been predicted by us in
earlier studies (2) and has since been confirmed experimentally (3). In
accordance with this, the average neck size developed between the particles
depends on the propensity of the liquid to wet the solid. When the overall
tendency for wetting decreases, the neck size developed between the particles
decreases.

Previous descriptions of LPS had conjectured that, excepting for micro-
structural coarsening, densification ceased and the structure is essentially a
static one following the development of a skeletal structure. We have shown
that this is not so. A skeletal structure develops early on in the sintering
process (1), but the structure retains many dynamic features even though the
kinetics of microstructural development are reduced following formation of the
skeletal structure. We have investigated (4) the continued development of
micro and macrostructure in LPS alloys at long sintering times. In particular,
the phenomena of compact "slumping", a feature common at long sintering times
in skeletal structures, has been studied. We have shown slumping is caused by
solid particle "extrication" from the skeleton. Particles so extracted, one
by one, fall (or rise, as the case may be) a distance comparable to the solid
interparticle spacing, and this process repeated many times leads to the
noticeable alteration in macroscopic shape of a LPS alloy. Particle extrica-
tion is effected by the dissolution of interparticle contacts either by an
Ostwald ripening process or a similar surface energy driven dissolution as a
result of surface topology in the vicinity of such a contact. Extrication
times have been measured for several systems and the times correlate with the
ratio of the interparticle contact radius to the particle radius (4). Thus
while "slumping" is driven by gravitational forces, the kinetics of it are
controlled by thermodynamic (as reflected in the neck size between particles,

i.e. the amount of material that must be removed for extrication) and ordinary chemical rate considerations (as reflected by the rate of chemical dissolution of the interparticle contacts). A recent publication dealt (4) with this phenomena. In it, as in here, the description of the process was couched in terms of LPS structures, but it appears the work may have ramification for a wide variety of solid-liquid two phase aggregates.

In earlier studies (5) we had investigated the coarsening behavior of LPS systems. Coarsening rates increase rapidly with increasing V_p in LPS alloys (5,6). The increase cannot be explained on the basis of several early theories (7,8) that considered the dependence of coarsening kinetics on the volume fraction of the coarsening phase. We had explained this behavior in terms of the effect that interparticle neck growth, taking place subsequent to a solid-solid contact being made, has on the reduction in solid-liquid inter-phase area. In this model, the contribution of coalescence to coarsening increases rapidly with V_p and the model was successful in simulating the experimental results (6). Indeed, as it now stands, this model has a pre-dictive capability. More recent theoretical work (9), however, has argued against the need to involve coalescence to explain the rapid increase in coarsening observed at high V_p. This Voorhees-Glicksman model, which consi-ders capillarity only as affected by particle curvature, is equally capable of predicting the V_p dependence of the coarsening rate constant for alloys for which we have data. Irrespective of which model is most appropriate, our studies have clearly shown the marked acceleration in coarsening which takes place in LPS materials containing a high volume fraction of solid phase.

One of the most intriguing aspects of development of LPS structures relates to the final stages of densification. Initial densification takes place rapidly and is certainly related to "primary rearrangement" by the

classical description of LPS which has been clarified further by recent studies (10-14). Densities on the order of 95% are so achieved routinely during LPS processing.

But it is the last stage of densification that is the most critical in providing for optimal final properties as even small (e.g. 2%) amounts of residual porosity can significantly and adversely effect materials properties, particularly mechanical ones. Because of this much recent effort has been expended on clarifying the mechanism responsible for elimination of such remnant porosity. Of necessity such treatments are conjectural and the one advanced by us (15) is no exception. In our treatment we suppose that pores (since they are at least partially filled with gas, even if not of the equilibrium pressure, ($p = 2\gamma/r$, where γ = pore surface energy and r = pore radius), they can also be considered bubbles) driven by gravity forces wend their way to the sample surface and this can be the mechanism by which full density is attained in a LPS material. For this to be a viable mechanism, we showed that the pore must remain isolated within the liquid phase. That is if they were to partially "wet" the solid, they would remain attached to it until coarsening caused a pore to reach a critical size at which a pendant drop could be detached from it. The droplet could then migrate to another particle, re-attach itself and the process repeated. Such a scenario could, in principle, lead to full densification. In practice it would not for the radius of a pendant drop formed thusly is large in comparison to a typical liquid channel or particle size in a LPS material (15). Thus pores like this remain included within the material and densification by the gravity driven pore migration mechanism cannot happen. If wetting of the solid by the pore does not take place, it appears also that there is only a small "time window" in which the pore migration mechanism can effect full density. That is, pore coalescence

and/or the development of a skeletal structure can lead to pore radii large in comparison to the liquid channels providing the pore escape path. As mentioned the pore migration densification mechanism is a speculative one. Yet it does seem to provide a mechanistic bridge to the phenomenological correlation between rapid densification and the "secondary rearrangement" observed in a host of LPS materials. The secondary rearrangement phenomena is associated with liquid penetration of solid particle grain boundaries (10-14). It appears reasonable that during this process a morphology propitious for the pore migration densification mechanism is produced temporarily.

Our studies on the structural aspects of LPS can be summarized thusly. We have suggested a speculative, but physically plausible, mechanism for the last stages of densification during LPS. We have been able to describe contiguity in terms of morphological (V_p) and surface energy considerations and the continued development of microstructure and macrostructure (including coarsening) in a semiquantitative and, in some cases, predictive manner.

B. <u>Processing, Structures and Properties or Heavily Cold Worked Metal Matrix Composites</u>

Heavily cold worked (deformation strains in the range of ca. 3.0 to 10.0) two phase metallic materials display remarkable strengths (16-20). Tensile strengths of them are typically well in excess of those predicted by a rule-of-mixtures approach which explicitly considers the expected strain hardening of the individual components. The degree of cold work required to produce large incremental strengthening depends strongly on the materials combination. For example, combinations of FCC metals and strong strain hardening BCC transition metals manifest appreciable strength increments at drawing strains of only 2.0-3.0 (16,19,24). Conversely, combinations of two FCC metals, which

individually exhibit saturation work hardening at large strains, must be cold worked to deformation strains on the order of 6-7 (17-21) before appreciable excess strength is noted in them.

In our investigations (21,22), we have studied several facets of the processing-structure-property relationship in these intriguing materials. We have found that heretofore assumed restrictions regarding the individual constituents inherent capacity for plastic deformation do not universally apply in their manufacture. For example, previous studies (16-20) focused primarily on composites containing two phases possessing extensive capacity for plastic deformation (e.g. Cu-Fe, Cu-Nb, Ag-Cu, etc.). We have found that composites containing one phase having little capability for plastic deformation in bulk form can be produced. Several examples are illustrated in Figs. 1-3. Chromium "whiskers", a constituent of a Cu-Cr composite which have been extracted from it, are shown in Fig. 1. This material was manufactured by swaging and drawing a 15 vol.% Cr-Cu casting. The as cast structure contained isolated dendrites of Cr and the inherent ductility of these single crystals, together with the stress state accompanying drawing and swaging, allowed them to deform commensurately with the matrix. The composite so produced displayed exceptional strength for a Cu base alloy (T.S $\cong$ 840 MN/m^2 = 122,000psi). When the volume fraction of Cr was increased to the extent that the Cr became a continuous phase (at a volume fraction of $\cong$ 0.20), a composite could not be fabricated from the casting, it fractured early on in the drawing process. Thus we conclude that if a "brittle" constituent can be incorporated in single crystal and isolated form into a ductile matrix, a suitable composite can be fabricated from such a combination.

Further evidence of the above principle is provided in Figs. 2 and 3. The former shows a composite containing ca. 8 vol.% W fibers in a Ni-W solid

Fig. 1.   Cr fibers extracted from a heavily cold drawn Cu-17 vol.% Cr alloy.
The fibers, ca. one μm in diameter, were present initially as
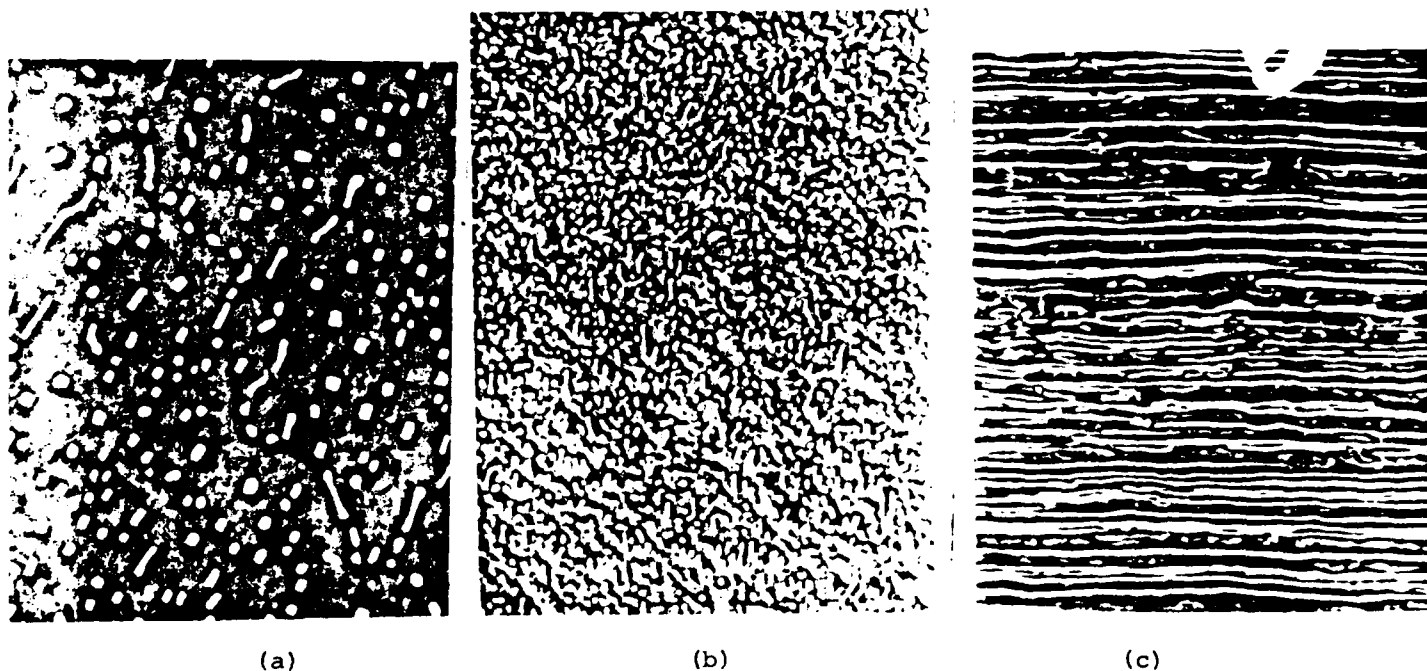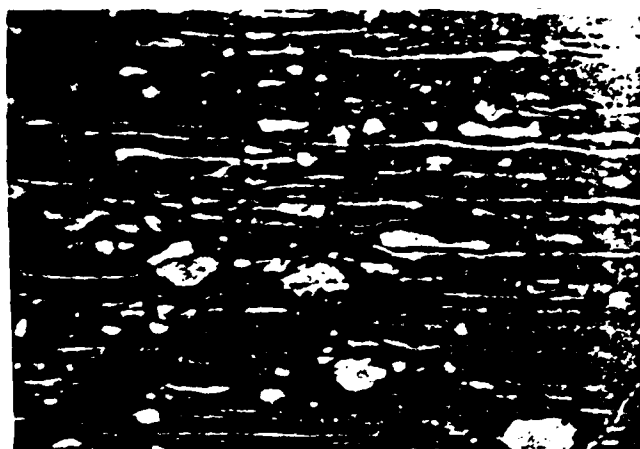isolated dendrites in a Cr-Cu casting.

(a)   (b)   (c)

Fig. 2.   Transverse sections of Ni-8 vol.% W euctectic alloy following (a)
directional solidification, (b) subsequent deformation to a drawing
strain of 2.1, (c) longitudinal section of b, illustrating the
fibrous nature of the W filaments. In (a) the W fibers have a
characteristic dimension of ca. 1μm. In (b) and (c), their size is
approximately an order of magnitude less than this.

solution matrix. In this instance, the starting structure was a directionally
solidified eutectic which consisted of aligned W single crystals dispersed
within the solid solution matrix. The as worked composite displays surpris-
ingly high strengths (ca. 240 MN/M^2 = 350,000psi) at moderate ($\varepsilon_T \cong 2.1$) drawing
strains. Figures 3a and 3b illustrated the "composite" structure developed in
a Cu-Mo alloy after mechanical drawing. This material was fabricated initially
by a solid state sintering process. During the early stages of drawing, the
polycrystalline Mo particles did not deform to anywhere near the degree the
matrix did (Fig. 3a). However, at greater drawing strains (Fig. 3b), the
molybdenum phase is beginning to develop a fibrous form. We believe the
development of microstructure in this material takes place in the following
way. During initial deformation it is clear that the molybdenum does not
deform plastically. After a certain degree of deformation, the stress
developed in the molybdenum is sufficient to fracture intergranularly the
originally polycrystalline particles. Subsequent to this particle fragmen-
tation, the so produced single (perhaps bi) crystals deform to the same degree
that the matrix does in a manner analogous to that manifested by the Cr
dendrites in the Cu-Cr alloy. The "key" to successful composite fabrication
in the Cu-Mo alloy is that the microcracks concurrent with particle fracture
do not propagate during the manufacturing sequence. This requires a "tough"
matrix and a molybdenum volume fraction sufficiently low so as to prevent a
high density of microcracks and molybdenum particle contiguity; both of these
characteristics will promote fabrication failures.

The several examples shown illustrate two important points. Firstly, a
variety of processing techniques are suitable for producing initial structures
which can be subsequently manufactured into high strength cold worked metal
matrix composites. Moreover, the initial processing techniques (e.g. casting

(a)   (b)

Fig. 3.   Longitudinal sections of drawn ((a):ε_T=3.0, (b) ε_T=7.2) Cu-11.3 wt.%
Mo composites. It is only after significant drawing strains have
been imposed that the Mo particles elongate in the draw direction.
The dimension of the "equiaxed" particles (a) is ca. 12 µm. The
thinnest "fibers" in (b) have a dimension of ca. 0.5µm.

or powder metallurgy) are well characterized as are the deformation processes employed subsequently. Thus these composites are potentially much less costly than many of the currently important metal matrix composites, many of which necessitate elaborate and expensive processing to produce them. Secondly, a variety of materials, including many which display "brittle" behavior in bulk form, can be incorporated into heavily cold worked metal matrix composites. By so doing, the inherently high strengths of them can be utilized in structural form.

The fundamental scientific question regarding these materials is what causes their high, sometimes remarkably so, strengths? Only two strengthening mechanisms seem possible; the microstructural refinement concurrent with deformation processing and/or some type of additional dislocation strengthening. It is inherently difficult to separate the contributions each of these might make to strengthening, for microstructural scale decreases concomitantly with continued deformation during processing. Nonetheless we believe the incremental strength displayed by cold worked composites are caused by additional dislocation density in them in comparison to that obtained in single phase materials subjected to the same degree of cold work. This deduction arises from several considerations. First, similar microstructural refinement takes place in single phase materials in the sense that individual grains in them are thinned in one (or two) dimensions. Moreover, "obstacle" spacings in heavily cold worked materials have dimensions less than the grain size; that is individual dislocations or cell walls composed of dislocations, rather than grain boundaries, are the obstacles to dislocation flow. We can also assert that the inherent strain incompatibility between grains in a two phase polycrystal is greater than it is in a single phase polycrystal. In the latter, the orientation differences between adjoining grains gives rise to strain

incompatibilities which are accommodated by geometrically necessary dislocations. These give rise to a grain size dependence of the work hardening behavior of single phase polycrystals. For reasons noted above, this effect is even more pronounced in two phase materials. Moreover, continuing increases in interphase boundary area with deformation strain provides an equally continuing increase in the number of such geometrically necessitated dislocations. Thus we conclude that the *refinement in microstructural* scale is necessary to <u>effect</u> the increase in strength of cold worked metal matrix composites, but it is <u>not</u> <u>the cause</u> of it; rather the high density of geometrically necessary dislocations is the source of the high strength in these materials.*

We have been able to quantify these arguments to a considerable extent. We have extended the formulation of Ashby (23) for the geometrically necessary dislocation density (ρ_G) to the case where grain shape is altered as a result of large deformation strains. We find (21) that ρ_G is related to deformation strain, ε, and interphase spacing, d, by:

$$\rho_G \sim [1-\exp[-\varepsilon/2]]/d. \tag{1}$$

For small drawing strains, $\rho_G \sim \varepsilon$ in accordance with the initial model of Ashby, but at large strains, ρ_G varies inversely with d. If the incremental strength is assumed to scale with $\sqrt{\rho_G}$, then it correlates also with $d^{-\frac{1}{2}}$; i.e. a Hall-Petch type relationship, consistent with much experimental data (19,24,25), is observed.

In our model we have to arbitrarily "partition" the geometrically necessary dislocations between the two phases. This leads necessarily to an adjustable

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*A caveat is necessary here. Our arguments seem appropriate for drawing strains $\lesssim 7.0$. Something "happens" when deformation strains of ca. 10-11 are applied to two phase metals. In particular, the incremental strength they exhibit increases dramatically when cold working to such extents is applied to them, and our understanding of this phenomena is still sketchy at best.

parameter in the model. However, the partitioning coefficient is expected in most cases to lie between two fairly narrow values. These extremes correspond to equal numbers of geometrically necessary dislocations or an equal density of the same in the phases. Performing this arbitrary partitioning allows experimental and predicted composite tensile strengths (the latter being, of course, a function of measured d and deformation strain) to be compared; examples are shown in Figs. 4 and 5 for several of the systems we have investigated. It is clear that the agreement between "theory" and experiment is quite good. What is not clearly evident from the figures or the above description of the analysis, is that the model allows for approximate estimation of the strengths achievable in a given system. For example, greatest incremental strengths are obtained when the two materials comprising the composite have inherently great differences in their flow curves. Of course application of this principle must be tempered by the requirement of fabricability which becomes increasingly difficult to satisfy as the materials flow characteristics diverge. The model also predicts greatest incremental strengths are obtained when the constituents comprising the composite demonstrate strong work hardening effects. This is so because in this circumstance the geometrical and statistical dislocations interact in a synergistic way so as to effect large total dislocation densities. Thus Fe and Cr are excellent candidate materials for cold worked metal matrix composites because of their high values of strain hardening coefficient. Conversely the "saturation" hardeners such as Ag and Ni are not so as the annihilation processes which yield saturation hardening act to remove statistical and geometrically necessary dislocations alike. The above arguments are in accord with experiment. For example, Ag-Cu (17) and Ag-Ni (21) composites must be cold worked to large amounts before appreciable incremental strength is manifested in them.
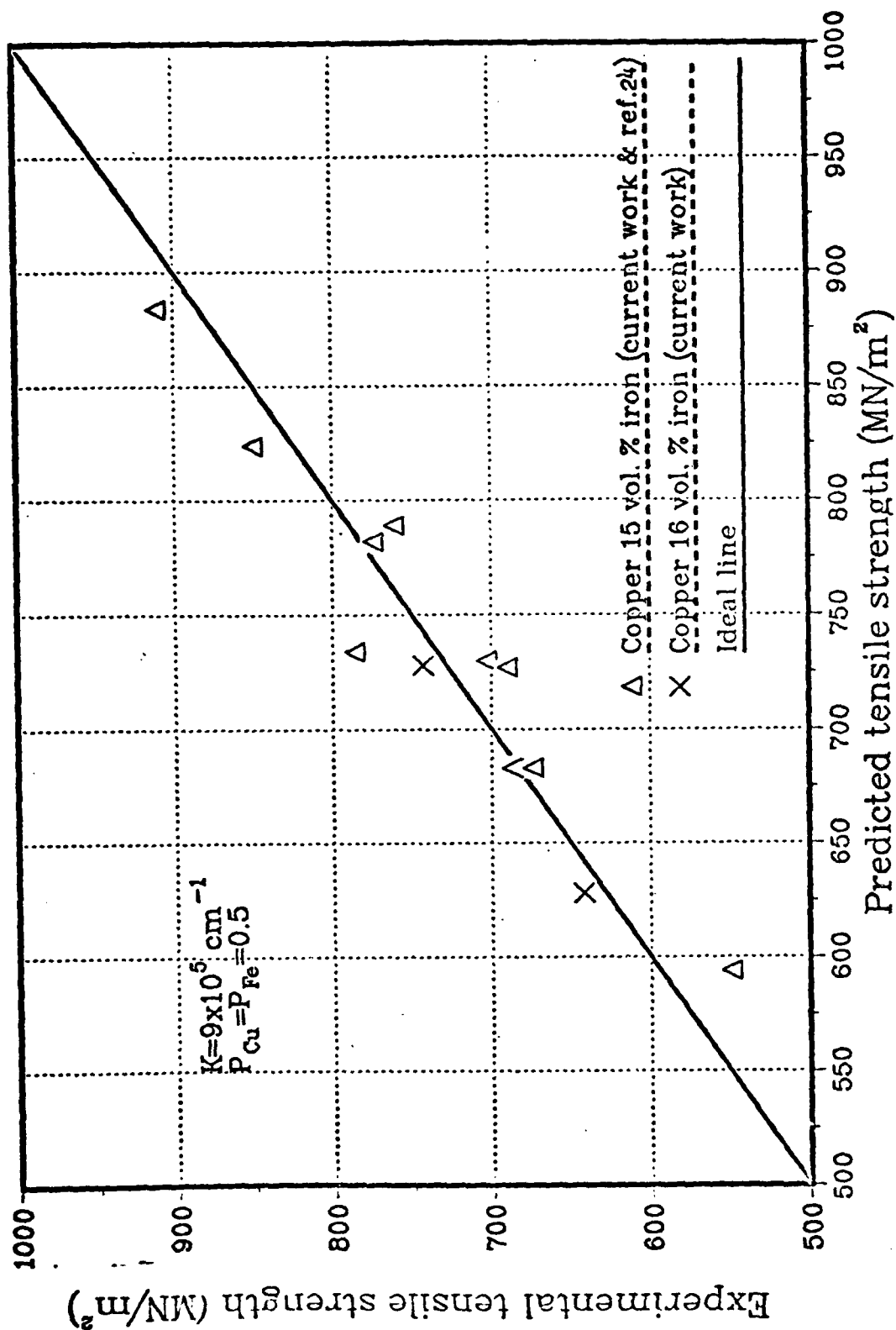
Fig. 4. Experimental vs. tensile strengths predicted on the basis of the geometrically necessary dislocation model for Fe-Cu composites. The value of P(=0.5) corresponds to partitioning of the necessary dislocations on a random basis between the two phases.
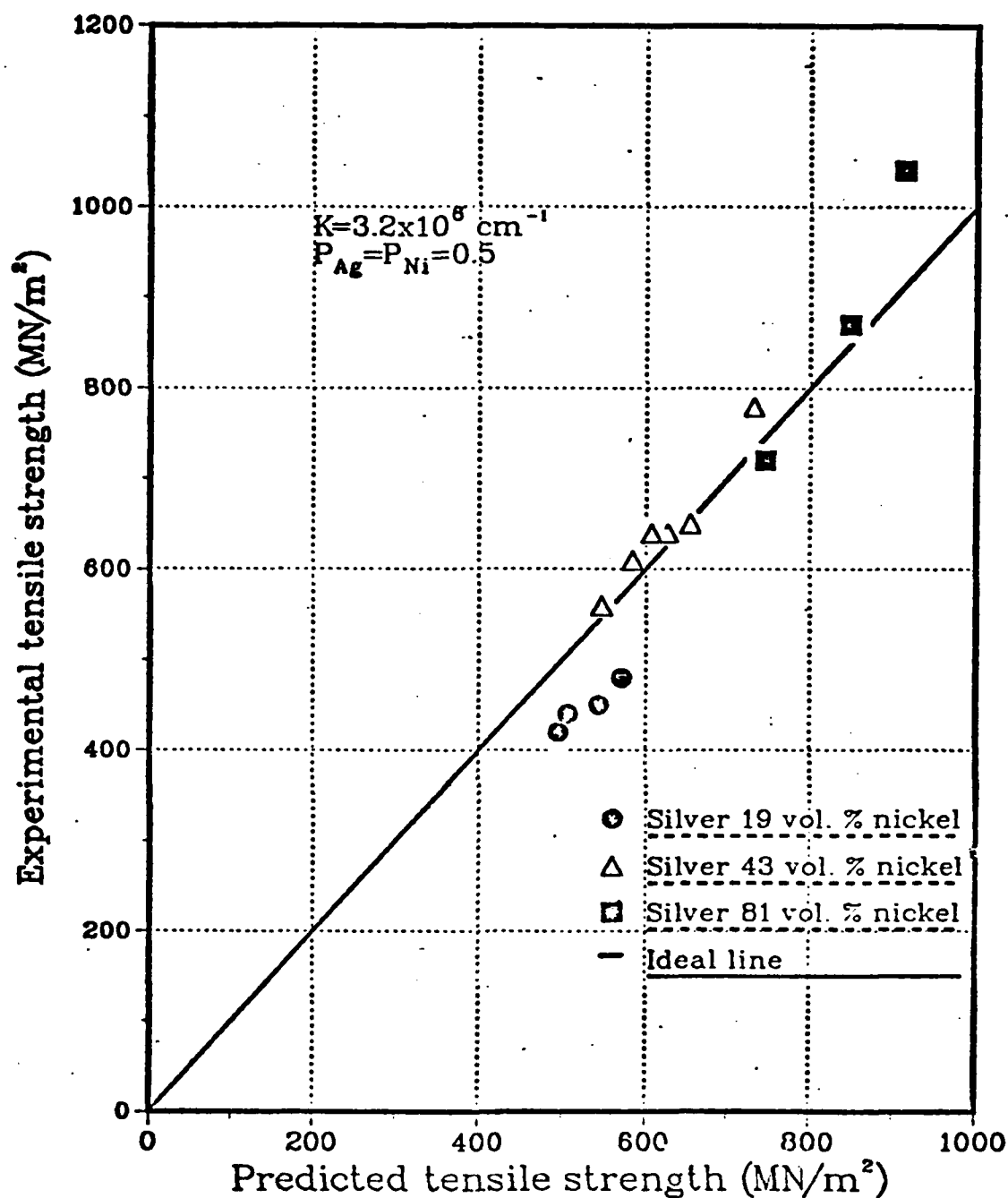
Fig. 5. Experimental vs. tensile strengths predicted on the basis of the geometrically necessary dislocation model for Ag-Ni composites. The value of P (=0.5) corresponds to random partitioning of the dislocations between the phases. That K for Ag-Ni is less than that for Cu-Fe (see Fig. 4) indicates that Ag-Ni composites display much less incremental strength than do Cu-Fe ones.

The structure developed in these in situ composites is also of interest. For example, isolated Cr and Fe particles in them develop ribbon like morphologies consistent with the plane strain deformation texture of the bcc elements. However, the interphase spacing between them in a composite develops in a manner consistent with the macroscopic axisymmetric deformation of wire drawing or swaging. Additionally, for Ag-Ni composites the macroscopic plane strain deformation of rolling also yields interphase spacings consistent with axisymmetric deformation (26). These features, of which we have no ready explanation, are shown in Fig. 6.

Progress in this area of research can be summarized thusly. We have been able to fabricate a number of heavily cold worked metal matrix in situ composites utilizing a variety of initial processing techniques. Moreover, in so doing, we have fabricated materials containing one phase which is incapable of extensive plasticity in bulk form. We have also been able to adequately model the strengths developed in these composites based on a geometrically necessary dislocation model. The model so employed leads to design criteria for these materials; viz. they should be as mechanically as disparate as possible subject to the fabrication requirements and should also manifest strong work hardening coefficients. Finally, the interphase spacing developed in these composites is consistent with that of axisymmetric deformation and this is so irrespective of macroscopically or microscopically imposed plane strain conditions.
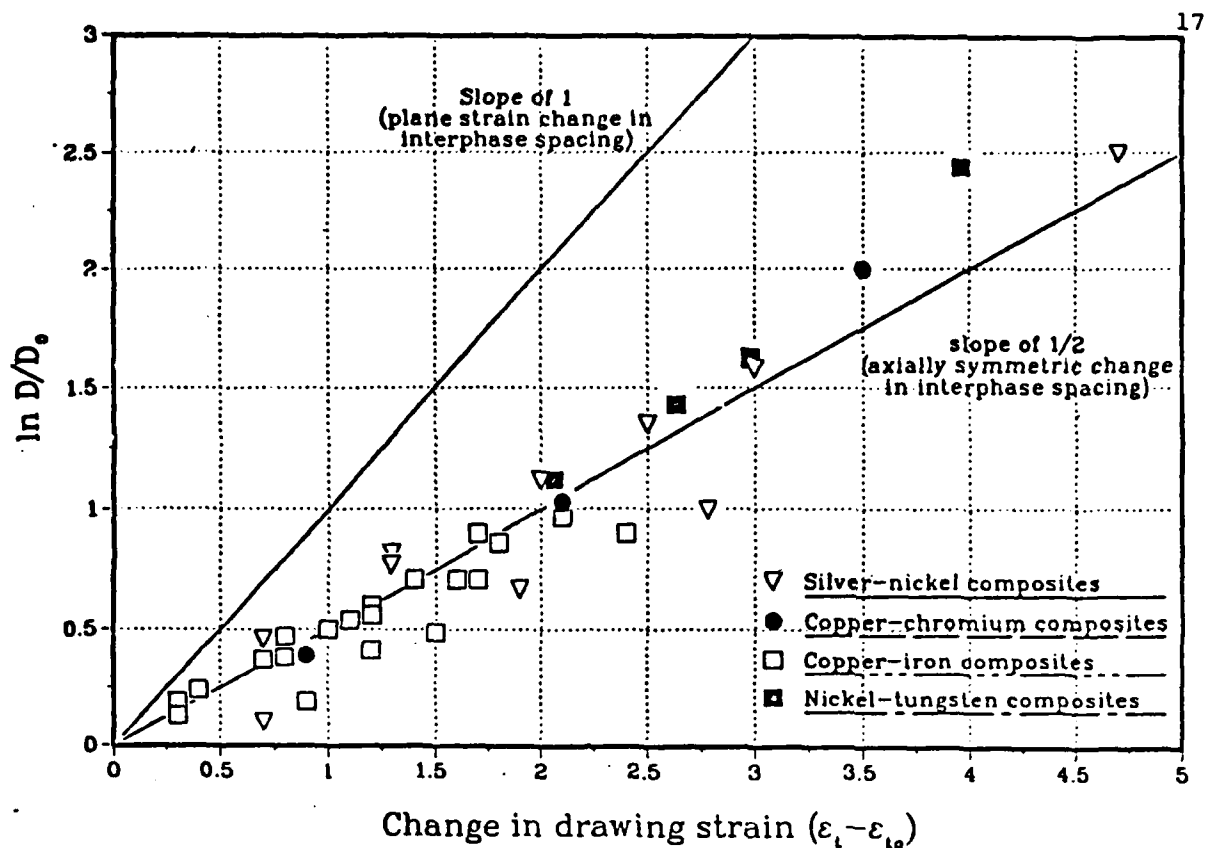
Fig. 6a. Change in interphase spacing vs. drawing strain for a number of mechanically fabricate: in situ composites. The morphological changes for the swaged and drawn alloys are consistent with an axially symmetric (slope = ½) change in interphase spacing regardle: of the microscopic shape changes effected.
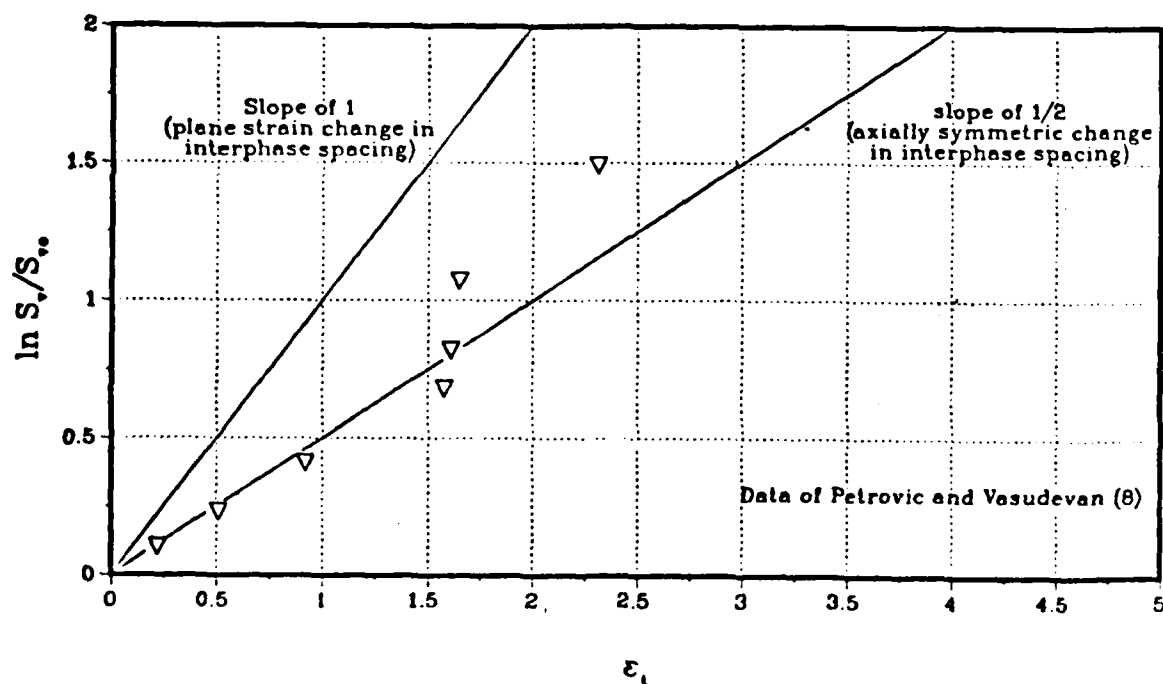

Fig. 6b. Change in interphase spacing of rolled Ag-Ni composites. Again the morphological change is consistent with axially symmetric deformation even though the macroscopic deformation mechanism is that of plane strain.

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Appendix A Publications (1981-) Resulting from this Grant

1. A. N. Niemi and T. H. Courtney, "Microstructural Development and Evolution in Liquid Phase Sintered Fe-Cu Alloys," J. Matl. Sc., 16, 1981, p. 226.

2. A. N. Niemi and T. H. Courtney, "The Temperature Dependence of Grain Boundary Wetting in Liquid Phase Sintered Fe-Cu Alloys," Metall. Trans. A, 12A, 1981, p. 1987.

3. P. D. Funkenbusch and T. H. Courtney, "Microstructural Strengthening in Cold Worked In Situ - 14.8 vol.%Fe Composites," Scripta Metall., 15, 1981, p. 1349.

4. A. N. Niemi, L. E. Baxa, J. K. Lee and T. H. Courtney, "Coalescence Phenomena in Liquid Phase Sintering -- Conditions and Effects on Microstructure," Mod. Dev. Powder Met., 12, 1982, p. 483.

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6. A. N. Niemi and T. H. Courtney, "Further Comments on Grain Boundary Wetting in Liquid Phase Sintered Fe-Cu Alloys," Metall. Trans. A, 14A, 1983, p. 977.

7. A. N. Niemi and T. H. Courtney, "Settling in Solid-Liquid Systems with Specific Application to Liquid Phase Sintering," Acta Metall., 3, 1983, p. 1393.

8. T. H. Courtney "Densification and Structural Development in Liquid Phase Sintering," to be published in a June, 1984 issue of Metall. Trans. A.

9. P. D. Funkenbusch, T. H. Courtney and D. G. Kubisch, "Fabricability and Microstructural Development in Cold Worked Metal Matrix Composites," submitted to Scripta. Metall.

10. P. D. Funkenbusch and T. H. Courtney, "Strengthening Mechanisms in Heavily Cold Worked Matrix Composites," in preparation.

Appendix B - List of Participating Scientific Personnel

1. T. H. Courtney, Professor of Metallurgical Engineering and Dean of the Graduate School.

2. J. K. Lee, Professor of Metallurgical Engineering.

3. A. N. Niemi, Ph.D. degree awarded 5/82.

4. P. D. Funkenbusch, Ph.D. degree awarded 5/84.

5. T. A. Nielsen, M.S. degree awarded 11/82.

6. J. Malzahn Kampe, current Ph.D. student.

7. D. G. Kubisch, current M.S. student.