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on

The Structure and Properties of Dental Amalgam Alloys Prepared With Controlled Packing Pressures

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U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

The Structure and Properties of Dental Amalgam Alloys Prepared with Controlled Packing Pressures

Abstract

Dental amalgam alloys were prepared using controlled variations in both packing pressure and alloy particle size. An attempt was made to find some relationships between the resultant changes in microstructure and the observed tensile strengths for each alloy. Higher packing pressures were found to be associated with lower mercury contents and lower void contents in the alloy specimens. Quantitative measurements on high-contrast photomicrographs indicated a variation in void content ranging from about 1% to 16% depending on packing pressure and alloy particle size range. The presence of voids apparently has a significant effect on the strength of dental amalgam. A comparison of conventional amalgams with amalgams prepared from spherical alloy particles demonstrates the superior adaptability and greater strength of the spherical alloys particularly at low packing pressures.

1. Introduction

Dental amalgams are basically silver-tin-mercury alloys which are prepared at room temperature by means of a diffusion reaction between silver-tin alloy particles and liquid mercury. Since a significant fraction of the original silver-tin alloy particles remains essentially unreacted, the result is an inhomogeneous structure made up of partially reacted silver-tin alloy particles embedded in a solid matrix of intermediate phases which contain most of the original mercury.

The microstructure of these alloys is affected by various factors such as the size and shape of the original silver-tin alloy particles, the amount of mercury in the alloy, the presence of cold work in the alloy particles, and probably, to some extent, by the surface condition of the particles.

In order to explore the relationships which may exist between the structure and properties of these alloys, it is desirable to produce controlled variations in the microstructure. The use of spherical alloy particles has enabled previous investigators to establish quantitative relationships between the average particle size and certain physical properties of these alloys [1]. In the present investigation dental amalgam alloys were prepared from spherical alloy powders with known particle size ranges and employing controlled variations in pressure during their condensation. An attempt was made to correlate the observed microstructures with experimentally determined values of tensile strength for these alloys. In addition, a comparison was made between the characteristics of dental amalgams prepared with spherical alloy powders and those produced from conventional alloy powders which have an irregular shape and contain wider variations in particle size.

2. Experimental Procedures

Tensile strength is probably one of the most important properties in clinical dental amalgam restorations [2] but previous investigators have usually employed the compressive strength of these alloys as an index of their utility under load.

Experimental values for compressive strengths, however, are apparently rather sensitive to the testing conditions and are particularly sensitive to the loading rate [3].

The values for tensile strengths probably provide a more reliable measure of the useful strengths of the alloys since these values are less sensitive to the rate of loading [4]. Furthermore, clinical fractures would perhaps be more likely to result from tensile loads than from compressive loads since the tensile strengths of these alloys are only about 1/5 as great as their compressive strengths. The tensile strengths were determined on small cylindrical specimens (approximately 4 mm diam. X 8 mm length) using the diametral-compression test recently adapted to the testing of small samples of brittle materials by Rudnick, Hunter, and Holden [5]. Burns and Sweeney have demonstrated that this method is reliable in determining the tensile strength of dental amalgam [6].

The tensile tests were conducted at a constant stress rate of 500 lbs/min and using a pad material of 0.005 inch aluminum foil to insure proper stress distribution. Each value reported is the average value obtained from six specimens.

The spherical alloy particles used in this investigation were from the identical lot used by Demaree and Taylor and were separated into particle size increments corresponding to those listed in their paper [1]. Two widely used conventional alloys, selected from the American Dental Association list of certified dental materials were also tested. Compositions are given in Table 1.

A standardized technique was followed in specimen preparation; the only variation being trituration time which was adjusted to suit the individual alloy. Trituration times are shown in Table 2.

A mercury-alloy ratio of 7/5 was employed using 0.84 ± 0.02 gm of mercury and 0.60 ± 0.01 gm of silver alloy particles in preparing each specimen.

The weighed mercury and alloy were placed in a plastic capsule and triturated in a low energy amalgamator operating at 3430 cycles per minute. Condensation of the triturated mass of amalgam was accomplished in a constant pressure packing device as described by Caul [3]. The load was applied for one minute beginning 30 seconds after the completion of trituration. Following removal of the load, any expressed mercury was brushed from the die and the specimen was ejected.

The packing device was calibrated against a compressive load cell to provide constant packing pressures of 95 psi, 268 psi, 535 psi, 1070 psi, and 2140 psi.

Although constant packing pressures were applied at the surface of each specimen it is questionable whether the same pressure existed at all points within the specimen. It is unlikely, however, that sharp pressure gradients would exist in materials containing large quantities of a liquid phase since hydrostatic loading conditions would probably predominate in such a material. Nevertheless, several specimens were sectioned longitudinally and transversely in order to examine their microstructures for any evidence of large pressure-gradient effects. In each of these specimens, metallographic examination revealed no visual evidence of variations in microstructure which might result from pressure gradients within the specimen. It was, therefore, concluded that, although pressure gradients may exist under the experimental conditions used, they are probably too small to have a significant effect on the structure and properties of these alloys within the limits of the experimental errors. Furthermore the emphasis in this investigation is placed less on the absolute values than on the relative changes which were observed as the packing pressure was varied in controlled increments. Seven specimens were prepared from each of the eight spherical and two conventional alloys at the five different packing pressures and were allowed to age at room temperature for one week. Six of these specimens were then used for tensile testing and the remaining specimen was mounted for metallographic examination.

Specimens were prepared for metallographic examination in the manner described by Wing [7]. They were mounted in cold-cure acrylic resin. The maximum temperature observed during the curing cycle of the mount material (61.7°C) was below that at which transformations occur on heating in dental amalgam as indicated by thermal expansion measurements [8].

Void measurements were obtained from high contrast photomicrographs of the as-polished specimens (Fig. 1,2,3). The photomicrographs were placed on an electronic scanner [9] which measured the area occupied by the voids relative to the total area scanned. By the Delesse principle, the volume percent of voids in the specimen is equal to the area percent on the photomicrograph [10]. After obtaining the void content data, the polished specimens were etched with the two stage etch used by Wing [7] and representative microstructures were photographed using a Bausch and Lomb research metallograph.

Mercury contents of the hardened amalgams were determined for two samples at each characteristic packing condition following the method of Crawford and Larson [11] except that the pulverized samples were held in a Vycor tube under a dynamic vacuum at temperatures between 500°C and 600°C.

3. Experimental Results and Discussion

The primary effect of increased packing pressure on the microstructure of the experimental alloys was to decrease the percentage of matrix phases present. This, of course, results in an alloy which contains an increased volume percentage of silver alloy particles. This effect was rather pronounced for the conventional dental amalgams prepared from irregularly shaped alloy particles (Fig. 4 and 5) but was not so prominent in amalgams prepared from spherical alloy powders.

Several factors apparently determine the volume percentage of matrix phases in the alloy and the percentage of silver alloy particles embedded in the matrix. At low packing pressures less mercury is expressed during packing (see Fig. 6 and Table 3) and consequently, there is more mercury available to participate in the reaction. Since most of the mercury is located in the matrix this would tend to produce an increase in the volume percentage of matrix phases. One may also observe that the alloy particles are not as uniformly distributed throughout the matrix at low packing pressures as they are at higher packing pressures. A comparison of Figure 7A through 7E reveals that the average inter-particle spacing is somewhat larger at the lowest packing pressures. There was no significant change in the average grain size of the matrix.

Conventional alloys apparently react with mercury to a greater extent and probably at a more rapid rate than the spherical alloy particles. This effect is probably related to the amount of cold-work in the alloy particles since the presence of cold-work may produce a considerable increase in the rate of solid state diffusion at low temperatures. It is believed, therefore, that the spherical alloy particles, which are relatively strain free [12], react to a lesser extent than the conventional alloy particles containing an appreciable amount of cold-work introduced during their preparation. It is difficult to evaluate these effects in the conventional alloy amalgams, however, due to the wide range of particle sizes present and their irregular shape which prevents any accurate determination of the total surface area available for the reaction.

Packing pressure also has an influence on the percentage of voids present in the specimens as can be seen in Figure 8. The reduction in void content with increase in packing pressure is relatively small for any individual alloy but wide variations in void content were observed among alloys having different particle size ranges (see Table 5).

A straight line may be drawn through the points relating tensile strength to void content (Figure 9) and this line would extrapolate to zero tensile strength at a void content of about 16%. If the reduction in tensile strength were directly proportional to a reduction in cross sectional area produced by the presence of voids, one would probably expect the line to extrapolate to a zero tensile strength at nearly 100% void content. It appears, therefore, that the effect of voids on the tensile strength of these dental amalgams is considerably greater than would be expected from a reduction in cross-sectional area alone. This is probably due to the existence of stress concentrations at each void [13]. Since a relatively large void content is observed in most of these alloys it appears that more attention ought to be directed toward a reduction of void contents in dental amalgam. The observed values for tensile strength associated with each packing pressure are listed in Table 4. In every case as packing pressure increases, there is a concomitant increase in tensile strength as shown graphically by the selected data in Figure 10. The tensile strengths of amalgams prepared from selected spherical alloy particles are retained to a greater extent at low packing pressures where the strengths of conventional alloys have begun to decrease rapidly.

The relationship between particle size and tensile strength is shown in Figure 11 for specimens packed at 2140 psi. The tensile strength increases rapdily with particle size to a maximum value of about 8000 psi for the particle size range of 8-14μ diameter. A further increase in particle size, however, is accompanied by a decrease in tensile strength. Values for the tensile strengths of commercial alloys A and B are shown for comparison.

It is interesting to compare Figure 11 with a graph of void content versus particle size for specimens packed at 2140 psi shown in Figure 12. The two graphs appear to be inversely related with the maximum strength at 8-15μ particle diameter corresponding to a minimum void volume in the same size range. A similar relationship can be seen for alloys packed at other pressures (Table 5). This suggests that the observed dependence of tensile strength on the average particle size may be largely attributed to a variation in void content. It is difficult to establish a quantitative relationship between void content and tensile strength, however, since the data for all alloys plotted in Figure 9 indicates that considerable scatter occurs. This scatter may be attributed to differences in the size and shape of the voids (Fig. 1,2,3) as well as to the difficulty of obtaining photomicrographs which are truly representative of the entire sample.

It should be noted that the percentage difference between the largest and the smallest particle diameters within each of the original particle size ranges vary widely as shown in Table 2. The efficiency of sphere packing, as measured by the volume of interstitial space available between the spheres, is determined by the size distribution of the spheres and by the relative number of spheres having each discrete size. It has not been feasible, in this investigation, to obtain a measure of the relative number of spherical particles of each size within the rather broad particle size classifications. The observed tensile strengths, therefore, cannot be regarded as characteristic of an alloy containing only particles of a single size corresponding to the average for each size range. Consequently all graphs that relate to particle size should be considered as having a validity which is limited by considerations of packing efficiency for the alloy particles (see Fig. 13 A-G).

It seems likely that the packing efficiency would have an influence on the volume percentage of matrix phases present. Since the matrix phases contain most of the mercury in these alloys one might, therefore, observe a variation in tensile strength with mercury content if the matrix volume has an important influence on tensile strength.

Inspection of the data in Tables 3 and 4, however, eliminates the possibility of a simple relationship between tensile strength and mercury content since tensile strengths varying from 970 psi to 7900 psi are obtained, by interpolation, for alloys having identical mercury contents (see Fig. 14). For a given alloy, however, a decreasing mercury content is accompanied by some increase in strength, but this effect apparently operates differently among the various alloys as shown in Figure 14. Obviously, there are other important factors which determine the strength of these alloys and it is impossible to establish a reliable and consistent correlation between any two of the simple parameters employed in this investigation. More basic information is needed on the mechanical properties of the individual phases, their surface energies and the grain boundary interactions in these alloys before significant progress can be made in understanding their behavior.

An effect of more immediate concern to the dentist is shown in Figure 15. This photograph shows three different alloy specimens prepared in a cylindrical die at 95 psi packing pressure and at the standard mercury/alloy ratio of 7/5. The use of this extremely low packing pressure permits a graphical demonstration of what is perhaps the most striking difference in behavior between amalgam specimens prepared from conventional alloy particles and those prepared from spherical alloy particles. Specimens A and B are conventional amalgams prepared from two of the most widely used commercial alloys and specimen 5 is an amalgam prepared from spherical alloy particles in the 30-50μ size range. The superior ability of the spherical alloy to conform or adapt to the shape of the cavity at low packing pressures may have great significance in clinical dentistry. This property frequently referred to as "adaptability", is difficult to measure quantitatively, but the remarkable "adaptability" of spherical alloys has already been noted in clinical trials [14]. In addition, the use of spherical particles permits an accurate control over particle size which cannot be accomplished for particles having an irregular shape. Control of particle size has considerable influence on properties which are of great concern to the dentist such as strength, setting time, carvability, mercury content, etc.

4. Conclusions

1. Variations in packing pressure produce changes in the microstructure of both conventional and spherical dental amalgams. An increase in packing pressure reduces the volume percentage of matrix phases and produces a more uniform spacing between the alloy particles. This is accompanied by some increase in tensile strength.

2. The microstructures of these alloys indicate that in the presence of excess mercury a more extensive reaction occurs in conventional alloy amalgams than for spherical alloy amalgams. This effect is probably related to the existence of cold-work in the conventional alloy particles compared to the relatively strain-free condition of spherical alloy particles, and perhaps also to the presence of a greater percentage of fine particles in the conventional alloys.

3. Both the mercury content and the void content of dental amalgams are decreased by higher packing pressures.

4. The percentage of voids present in dental amalgam ranges from about 1% to 16% and values may depend to a varying extent on the packing pressure, particle size range, and mercury content. Voids are generally isolated in the microstruture at all packing

pressures.

The presence of voids reduces the effective cross-section of each specimen and this, together with a stress-concentration effect probably accounts for the observed variation in tensile strength with void content.

Variations in the void content seem to be largely responsible for an observed relationship between tensile strength and average particle size.

5. Mercury content alone does not determine tensile strength. However, if the particle size and shape are held constant, somewhat higher strengths are associated with lower mercury contents but the extent to which each alloy is affected by mercury content varies.

6. Dental amalgams prepared from spherical alloy particles are as strong or stronger than conventional dental amalgams and may be condensed satisfactorily at much lower packing pressures. The spherical particle amalgams also exhibit superior "adaptability" in conforming to the shape of a cylindrical cavity and they retain their strength at low packing pressures where the strength of conventional amalgams decreases rapidly.

The authors wish to thank Mr. John Turner of the American Dental Association for his work in determining the mercury contents of the amalgam specimens and Mr. Leonard Cann of the National Bureau of Standards for his assistance in determining the void contents.

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Table 1

Chemical Composition of Amalgam Alloys Used

Alloy	Composition %			
	Ag	Sn	Cu	Zn
Experimental*	70.6	26.1	2.5	0.4
Alloy A*	70.9	25.8	2.7	0.9
Alloy B*	70.2	25.9	3.7	0.1

* Data obtained by Dental Research Section, National Bureau of Standards

Table 2

Trituration Times and Size Variation within Each Size

Alloy	Trituration Time (Sec.)	Size Difference (%) $\frac{D_2 - D_1}{D_1}$ X 100*
Spherical 1-4μ	90	300%
Spherical 4-8μ	45	100%
Spherical 8-14μ	45	75%
Spherical 14-30μ	20	114%
Spherical 30-50μ	20	67%
Spherical 50-74μ	45	48%
Spherical 74-105μ	45	41%
Spherical 105-149μ	45	42%
Alloy A	20	-
Alloy B	60	-

* Within each size range particles of largest diameter D_2 - are larger than particles of smallest diameter D_1 by $\frac{D_2 - D_1}{D_1}$ X 100%

Table 3

Mercury Content of Hardened Amalgam Specimens Prepared with Different Packing Pressures

Each Value Reported is the Average of Two Determinations

Alloy		Condensation Pressure (PSI) 95	268	535	1070	2140
Spherical	0-4μ	59.3	57.6	55.6	53.6	50.5
"	4-8μ	60.2	58.9	58.0	55.0	54.8
"	8-14μ	57.8	54.5	52.4	51.4	49.5
"	14-30μ	55.2	53.2	51.0	49.1	47.4
"	30-50μ	54.7	53.3	52.6	51.6	50.4
"	50-74μ	52.0	50.1	49.4	48.7	48.0
"	74-105μ	51.4	50.4	49.5	48.6	47.7
"	105-149μ	52.3	50.8	49.9	49.5	48.3
Alloy A		57.3	55.9	53.8	51.7	48.4
Alloy B		58.1	57.5	55.8	52.6	49.5

Table 4

Tensile Strength (PSI) for Amalgam Specimens Prepared with Different Packing Pressures (Standard Deviations)

Alloy		Condensation Pressure (PSI) 95	268	535	1070	2140
Spherical	0-4μ	2820(429)	3710(161)	3750(290)	4090(223)	4200(313)
"	4-8μ	4100(856)	4820(496)	4490(600)	6540(532)	6050(817)
"	8-14μ	5620(810)	5990(576)	6800(651)	7860(416)	7960(625)
"	14-30μ	5430(472)	5580(773)	6513(686)	6670(621)	7130(524)
"	30-50μ	6240(487)	6680(460)	6870(388)	6740(395)	6970(416)
"	50-74μ	4830(643)	5690(336)	5960(336)	6400(318)	6110(843)
"	74-105μ	1790(211)	1810(117)	2040(106)	1890(254)	3000(292)
"	105-149μ	960(74)	970(93)	1050(62)	1100(94)	1130(66)
Alloy A		5180(491)	6430(441)	6240(339)	7390(661)	7430(939)
Alloy B		3230(969)	5210(355)	6730(1130)	7120(536)	7390(870)

Standard deviations were calculated using the formula:

$$S.D. = \sqrt{\frac{\Sigma(X-\bar{X})^2}{N-1}}$$

S.D. = Standard Deviation
where X = Value for a single specimen
$\bar{X}$ = Average value for N specimens
N = Number of specimens

Table 5

Void Content of Hardened Amalgam Specimens Prepared with Different Packing Pressures

Alloy		Condensation Pressure (PSI)				
		95	268	535	1070	2140
Spherical	0-4µ	15.42	12.04	12.72	8.42	7.52
"	4-8µ	3.46	7.77	5.54	3.65	3.17
"	8-14µ	3.90	4.25	3.79	4.70	2.31
"	14-30µ	8.52	6.67	6.39	6.09	4.95
"	30-50µ	5.32	3.92	2.87	3.97	4.25
"	50-74µ	11.14	6.29	9.07	6.60	7.02
"	74-105µ	12.43	10.87	10.64	10.65	10.61
"	105-149µ	13.04	11.26	11.10	13.29	11.22
Alloy A		8.98	6.28	6.04	5.80	2.68
Alloy B		2.61	1.31	1.00	0.69	0.50

Figure 1. Spherical Alloy Amalgam 0-4μ Size Range Prepared at 2140 PSI. Packing Pressure. Specimen is in the "as polished" condition. 125 X.

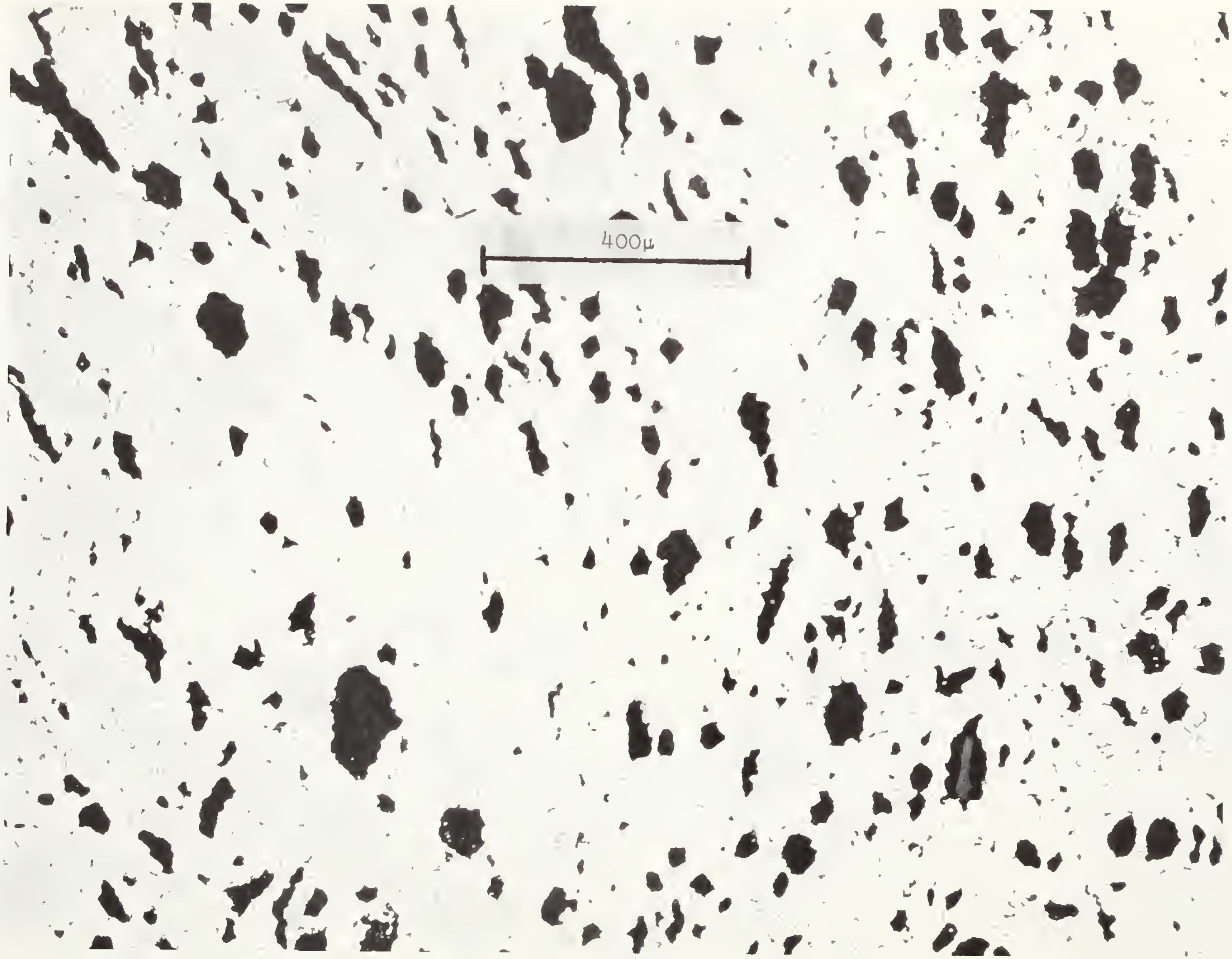

Figure 2. Spherical Alloy Amalgam 0-4μ Size Range Prepared at 535 PSI Packing Pressure. Specimen is in the "as polished" condition. 125 X.

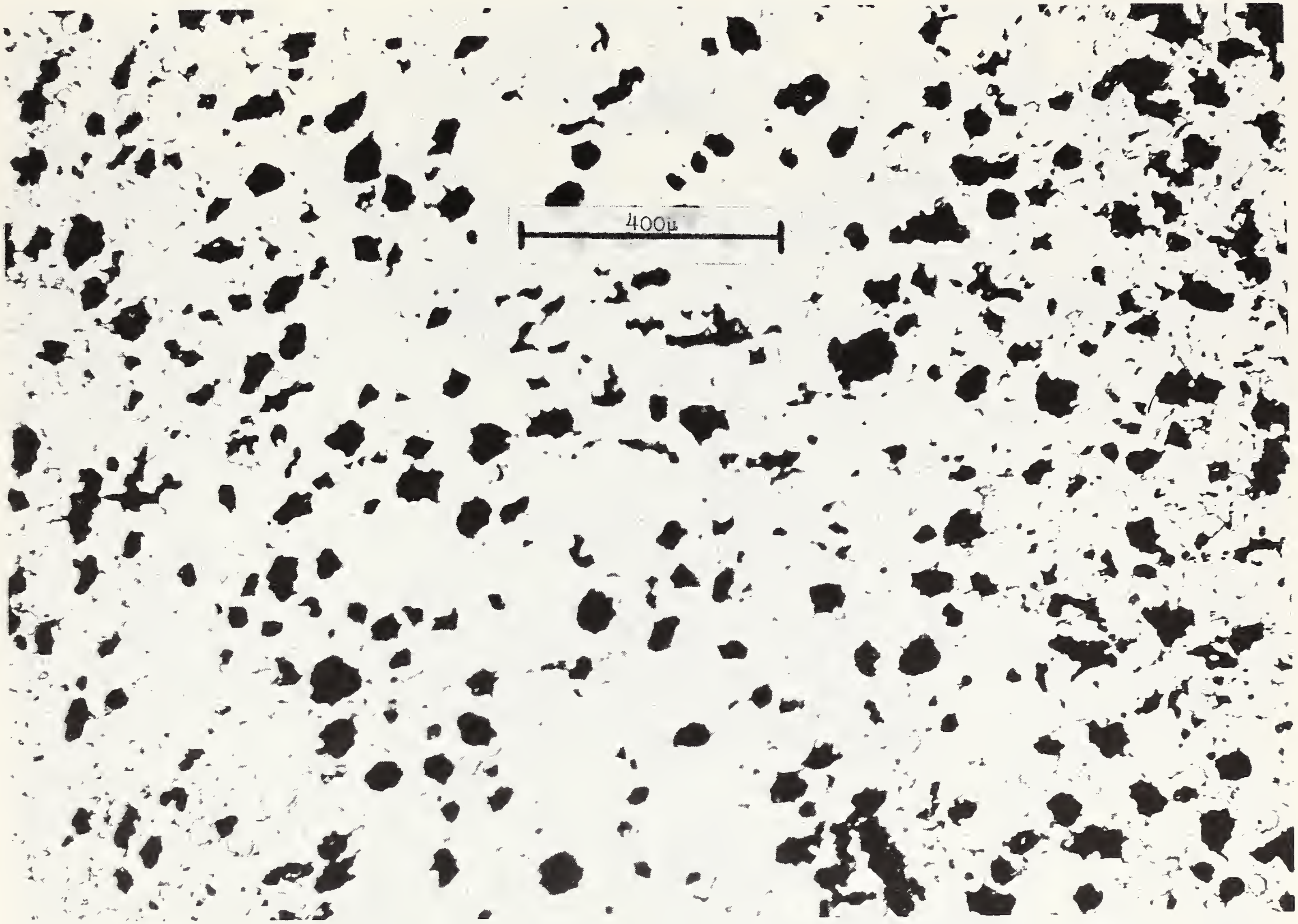

Figure 3. Spherical Alloy Amalgam 0-4µ Size Range Prepared at 95 PSI Packing Pressure. Specimen is in the "as polished" condition. 125 X.

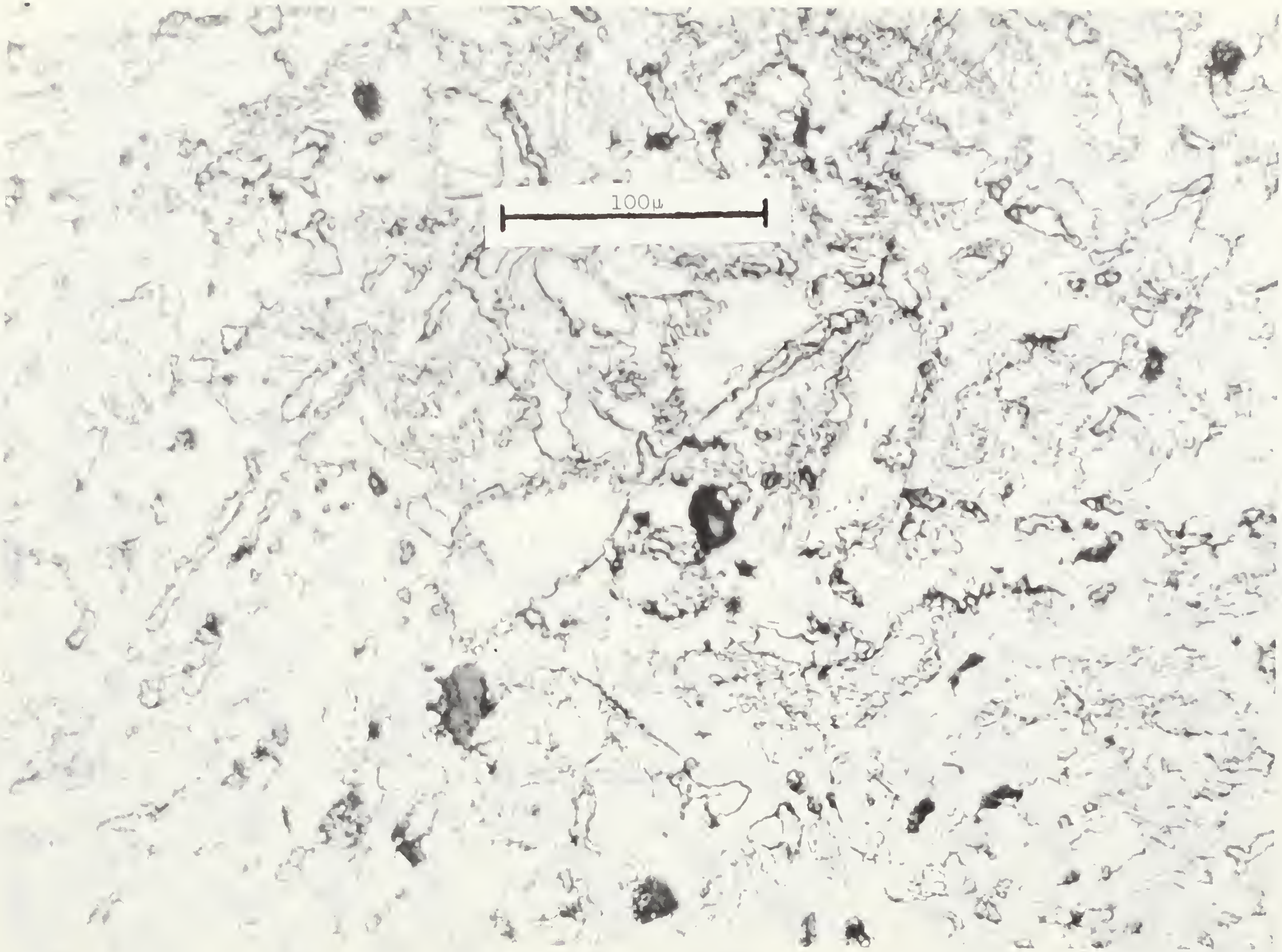

Figure 4. Conventional Alloy A Prepared at 1070 PSI. KCN + I_2 Etch 500 X. Mercury content is 51.7%.

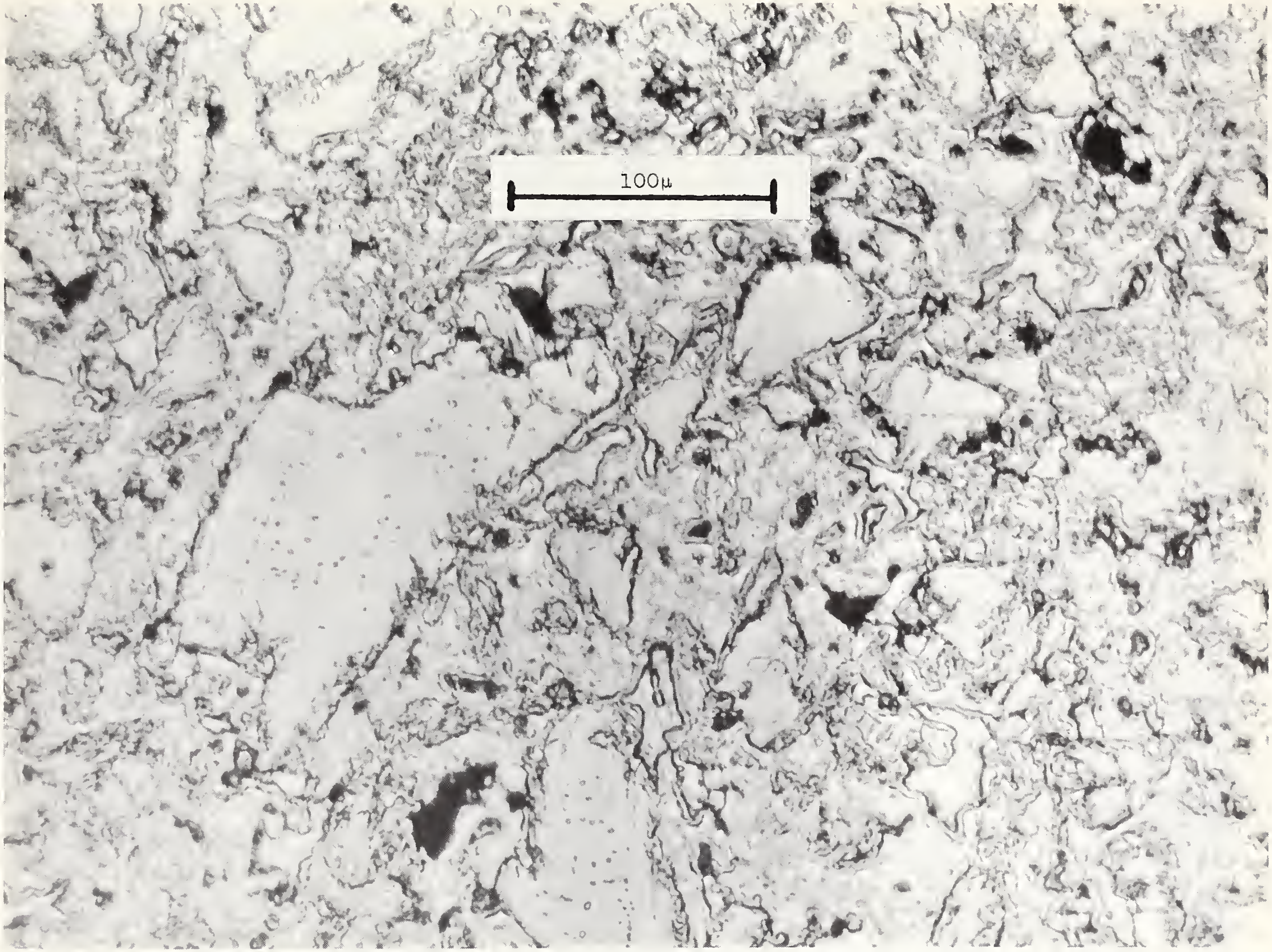

Figure 5. Conventional Alloy A Prepared at 2140 PSI. KCN + I_2 Etch
500 X. Mercury content is 48.4%.

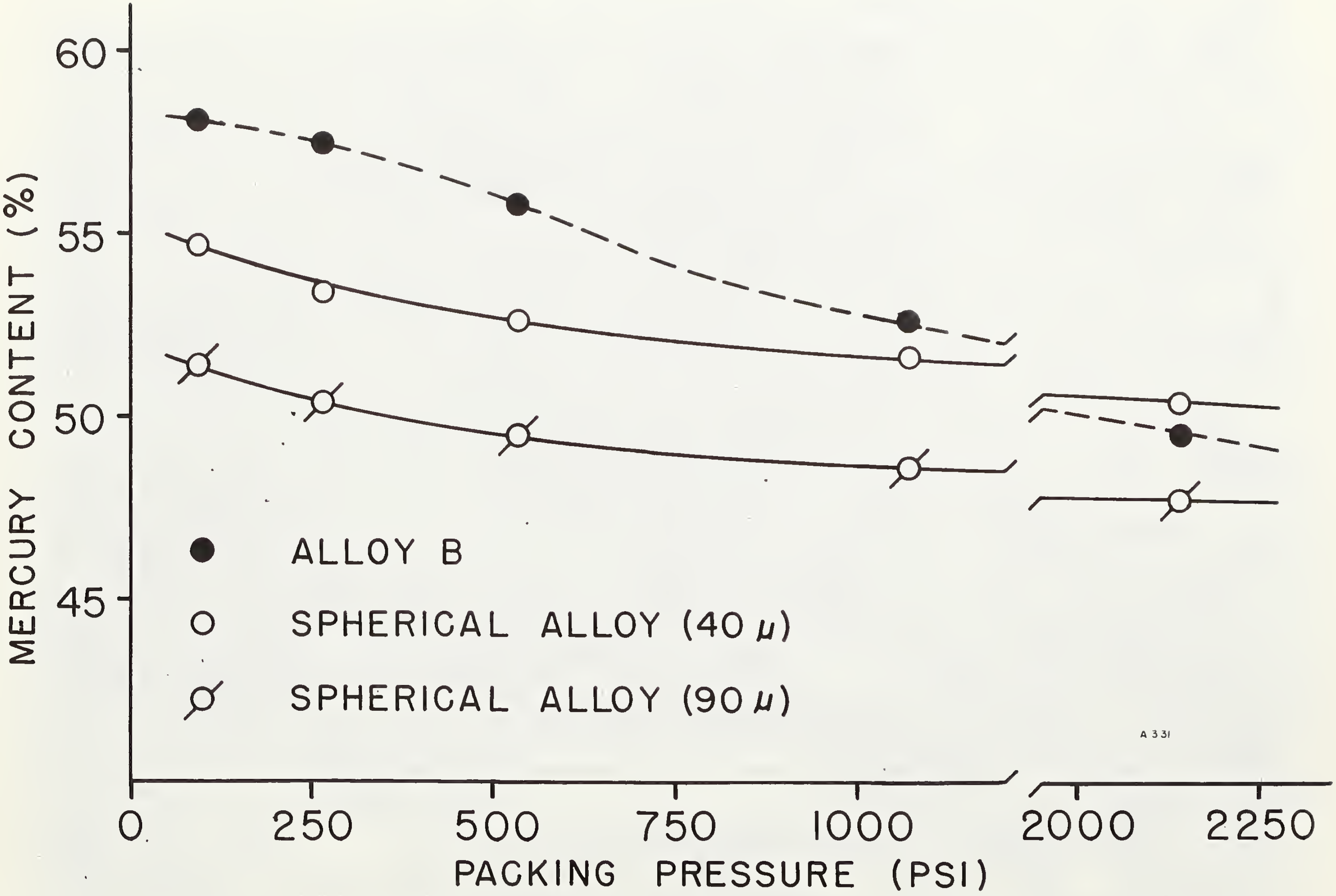

Figure 6. Packing Pressure vs Mercury Content. (Average particle sizes are given).

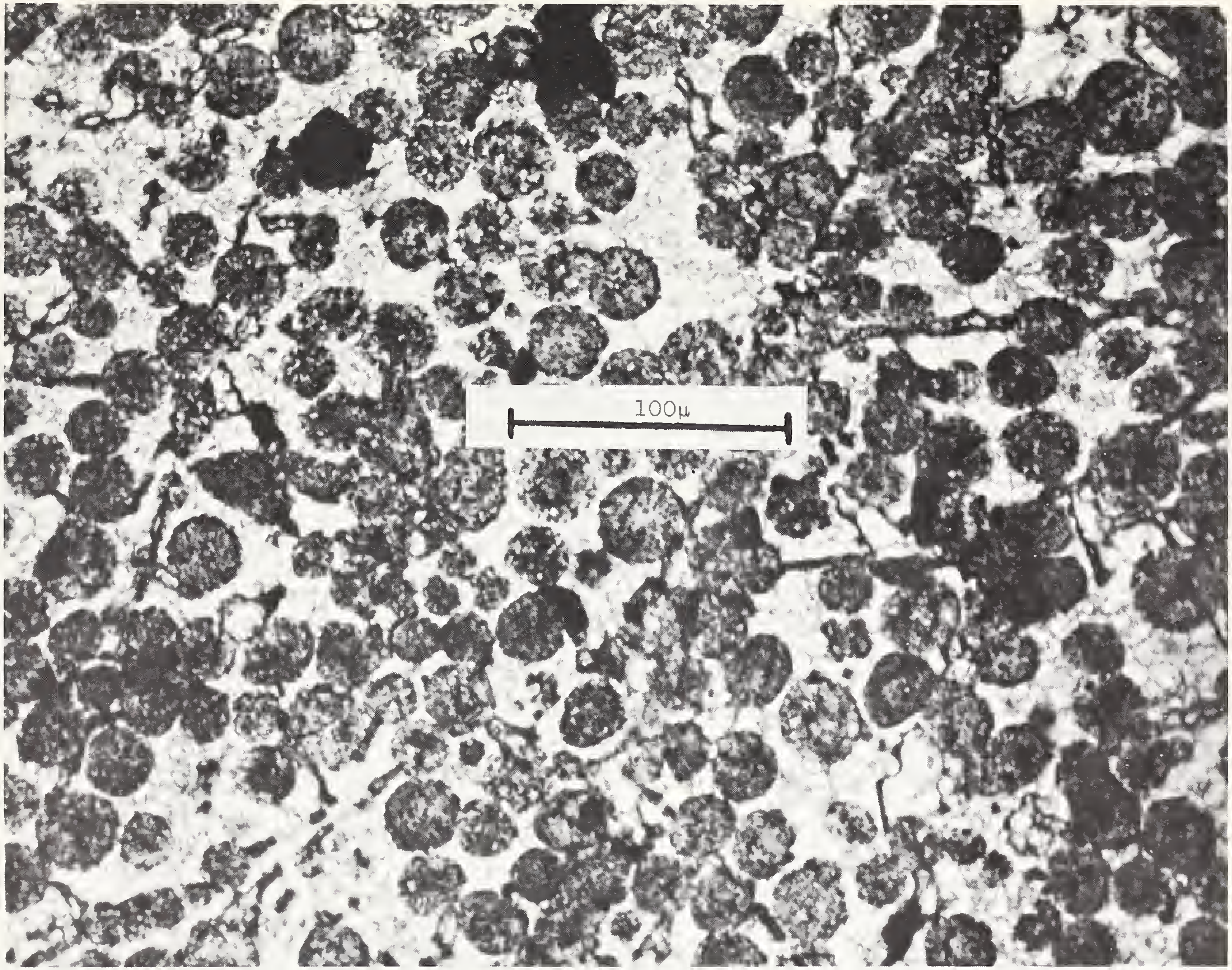

Figure 7-A. Spherical Alloy 14-30μ Size Range Prepared At: 95 PSI Packing Pressure.

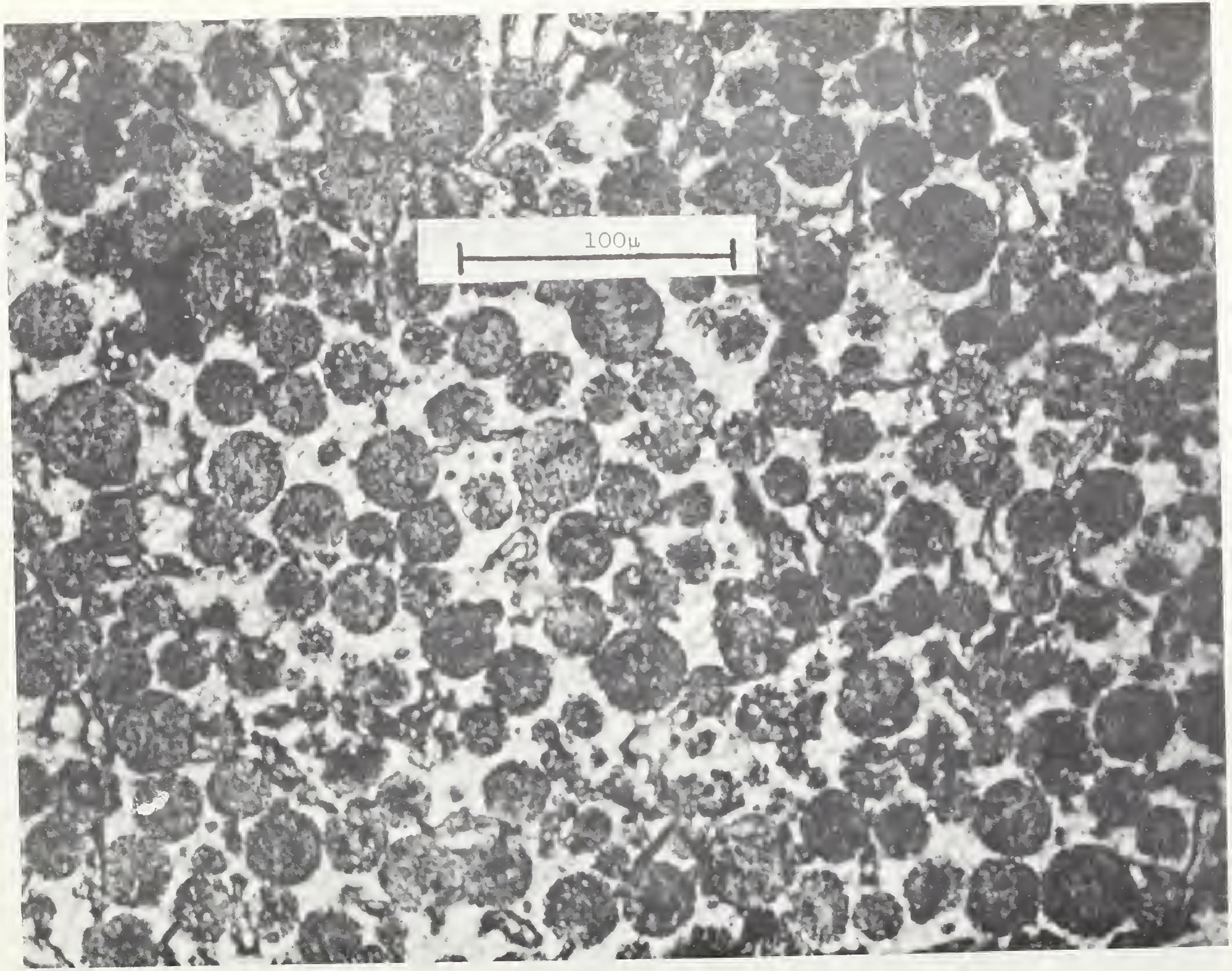

Figure 7-B. Spherical Alloy 14-30µ Size Range Prepared At: 268 PSI Packing Pressure.

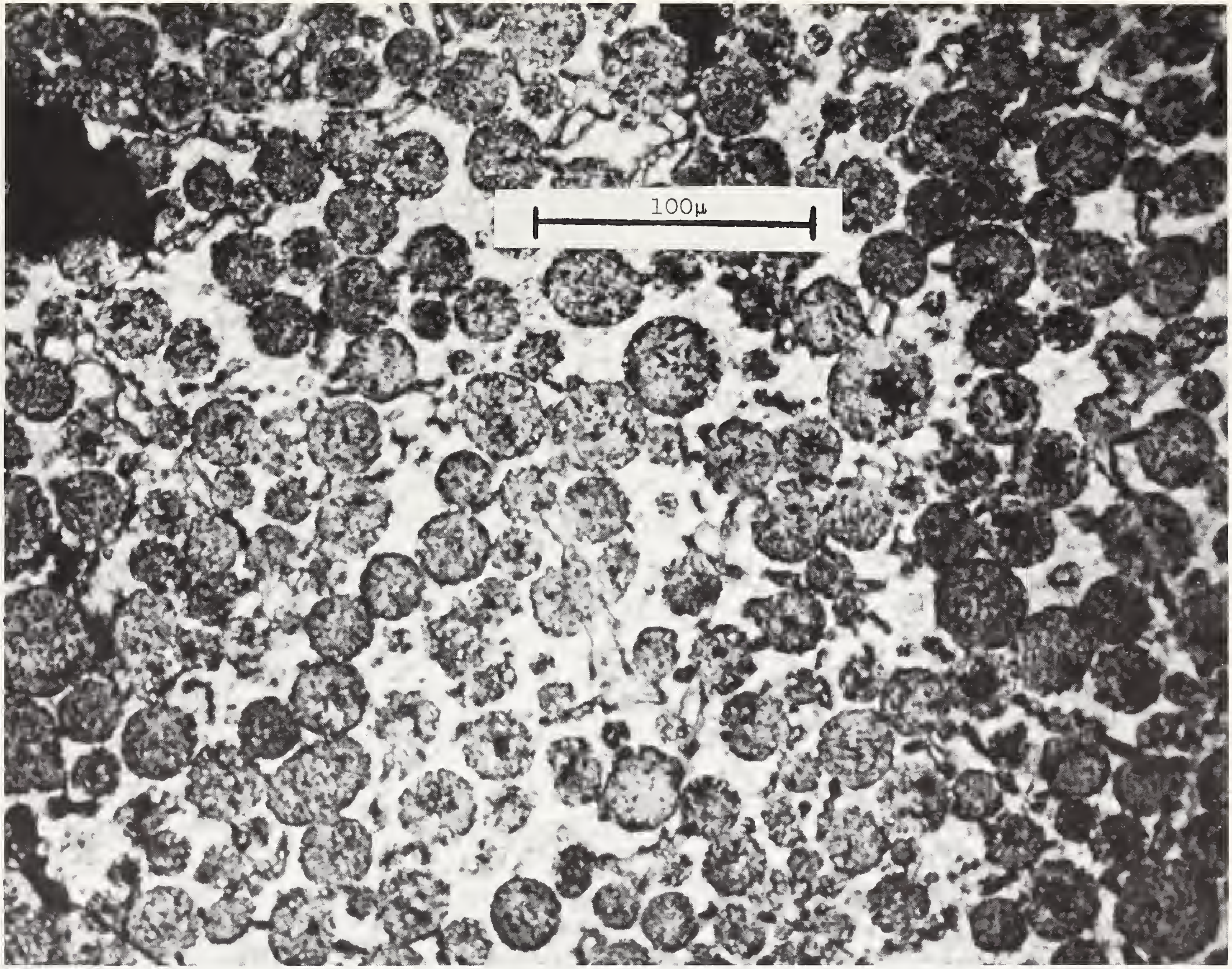

Figure 7-C. Spherical Alloy 14-30µ Size Range Prepared At: 535 PSI Packing Pressure.

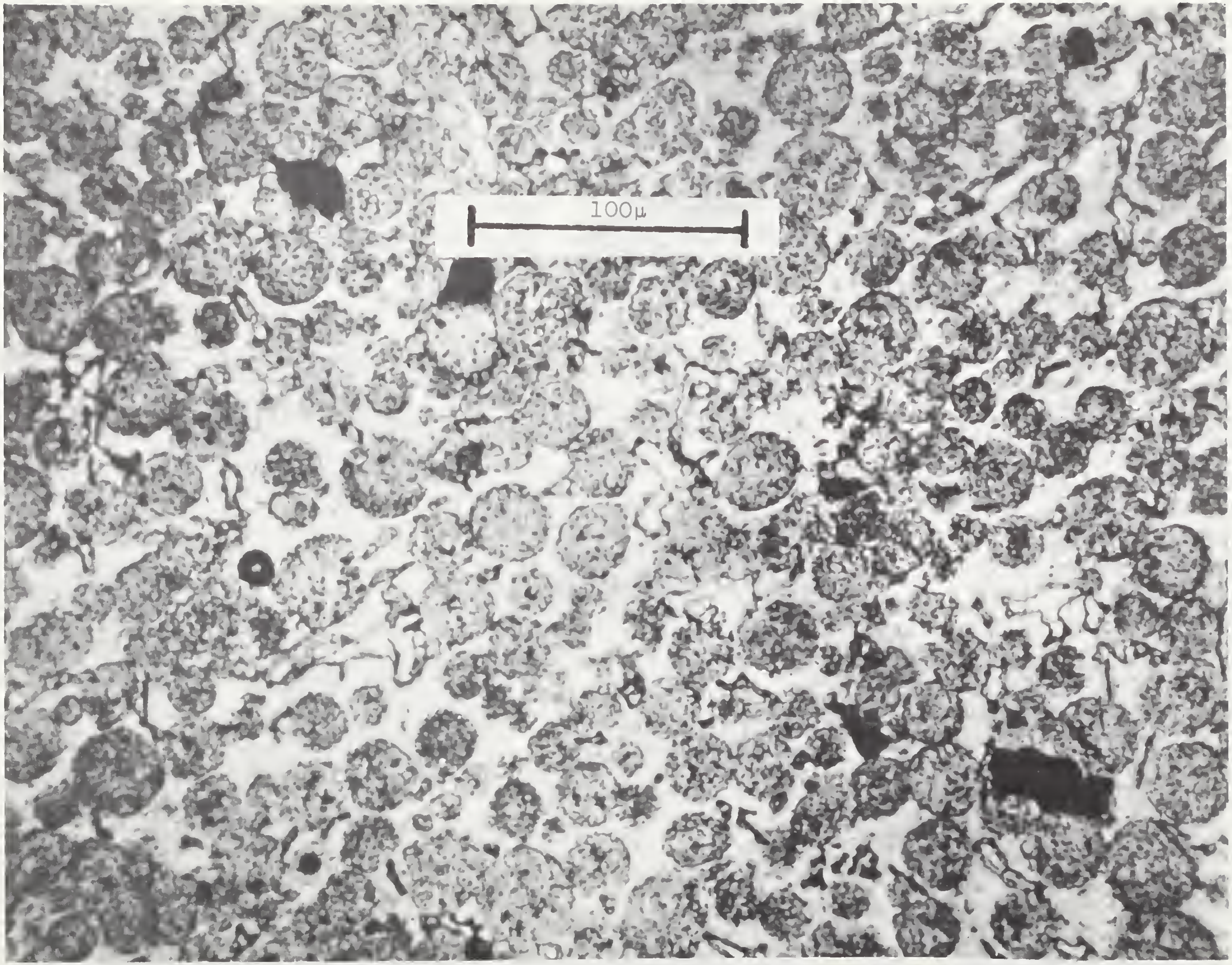

Figure 7-D. Spherical Alloy 14-30μ Size Range Prepared At:
1070 PSI Packing Pressure

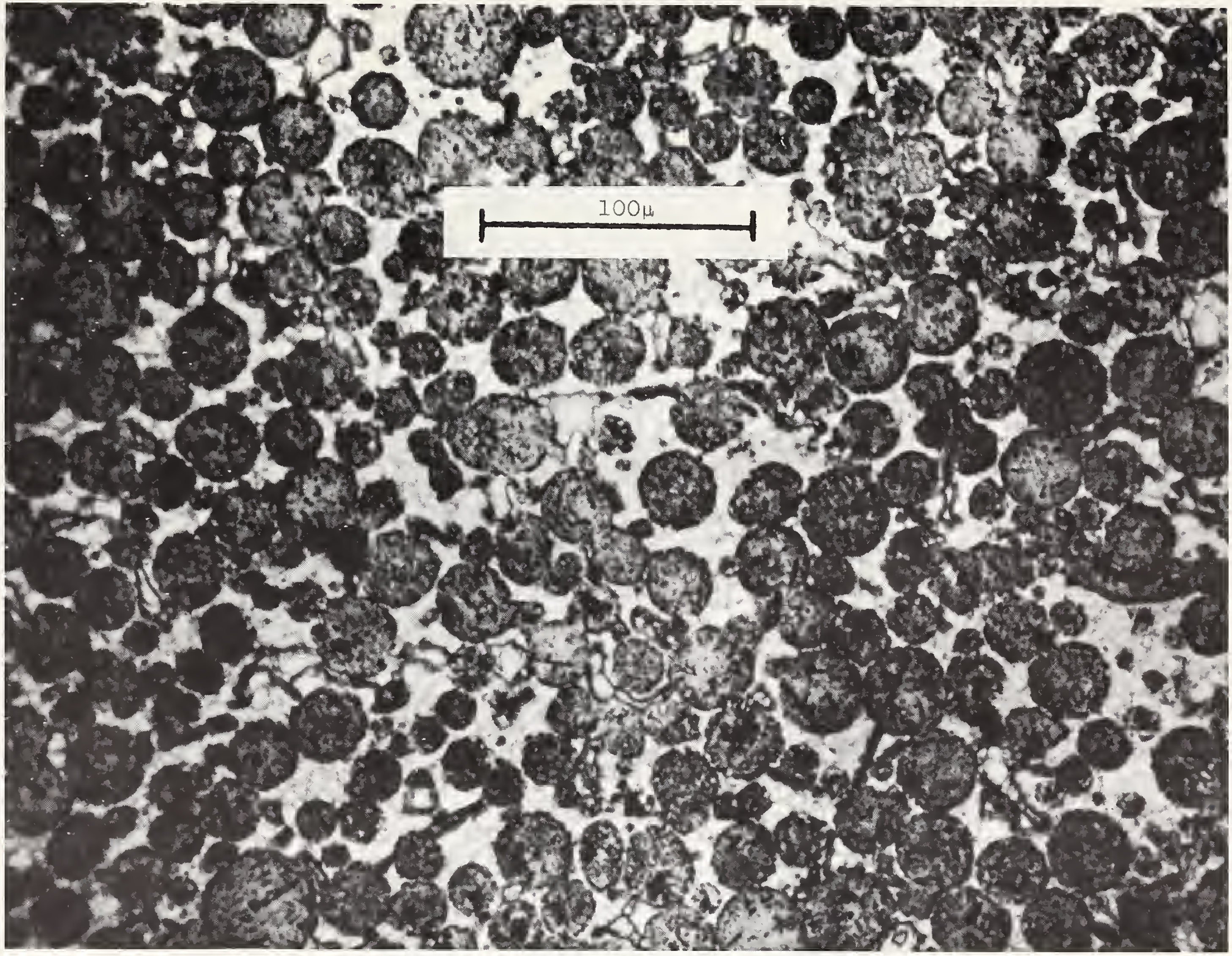

Figure 7-E. Spherical Alloy 14-30μ Size Range Prepared At: 2140 PSI Packing Pressure.

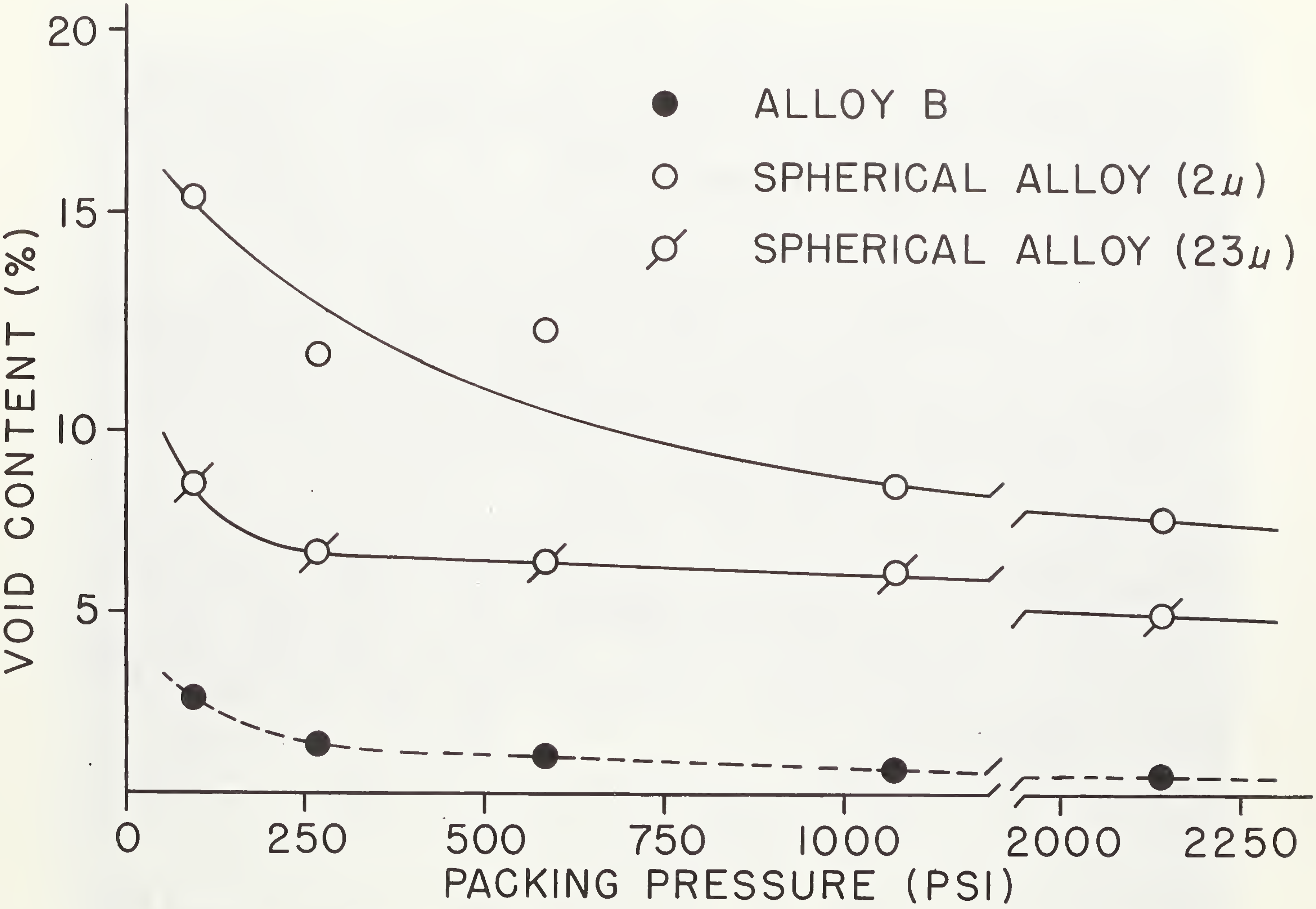

Figure 8. Packing Pressure vs Void Content. (Average particle sizes are given).

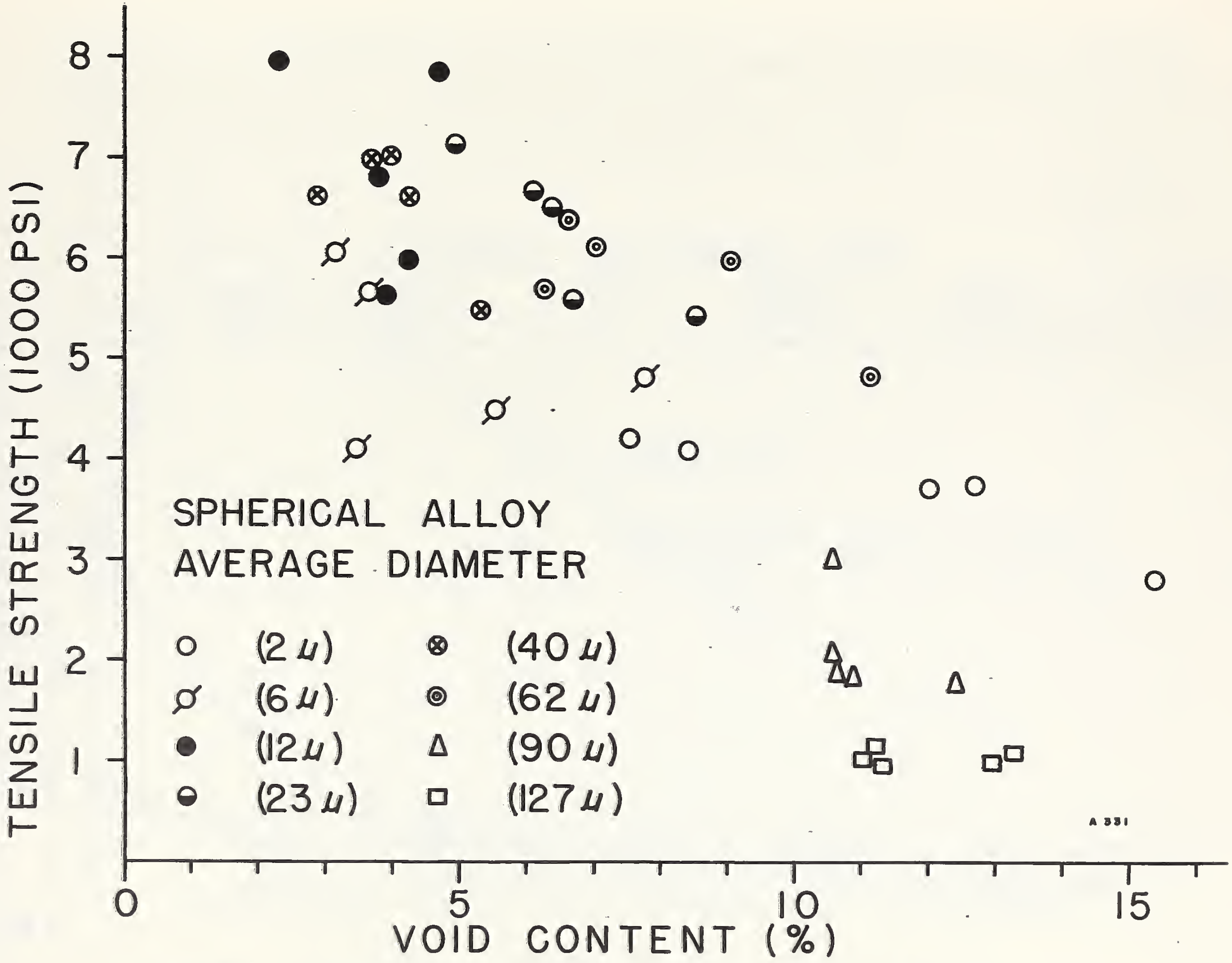

Figure 9. Void Content vs Tensile Strength for all Alloys Tested. (Average particle sizes are given).

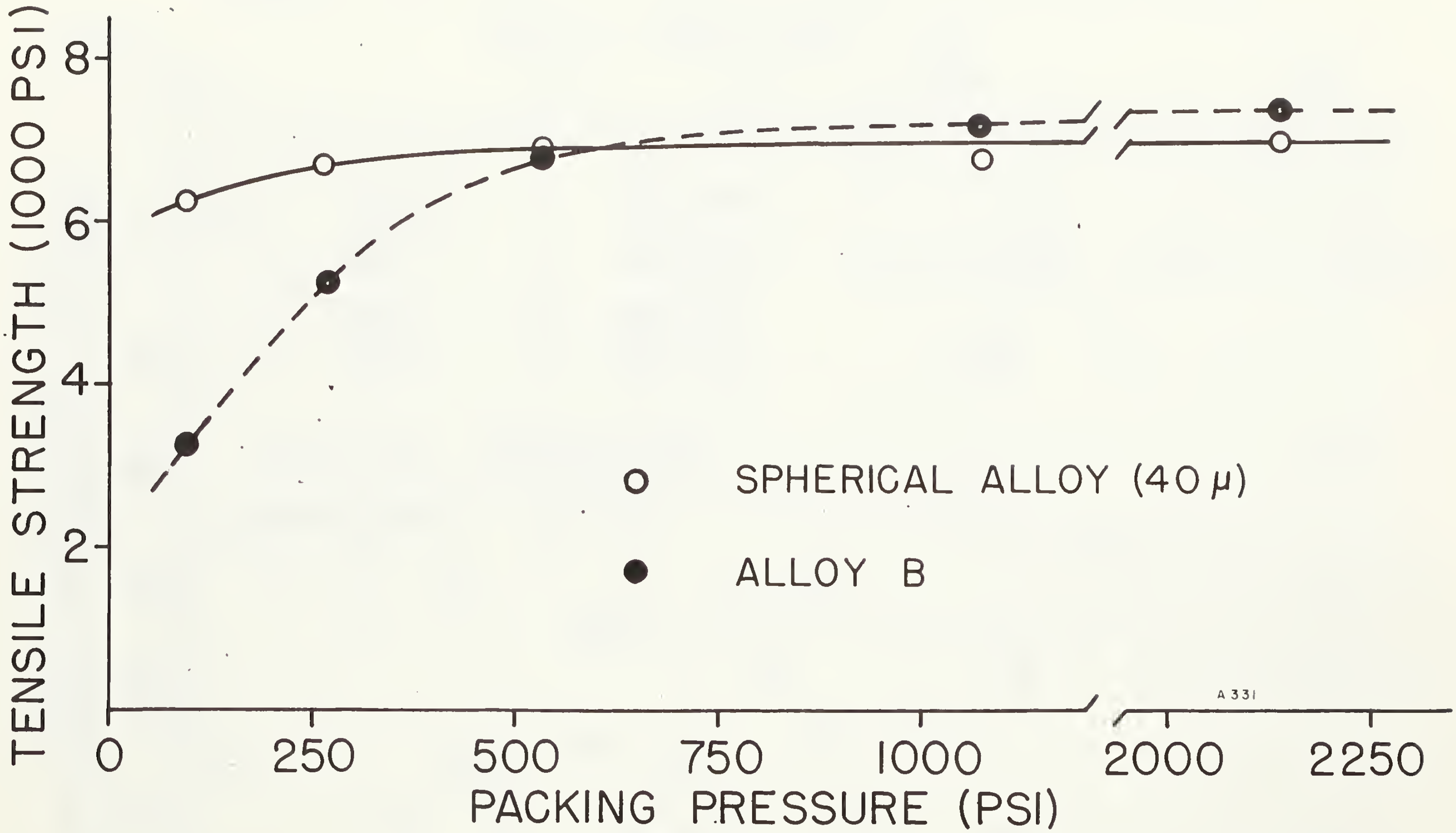

Figure 10. Packing Pressure vs Tensile Strength. (Average particle size is given).

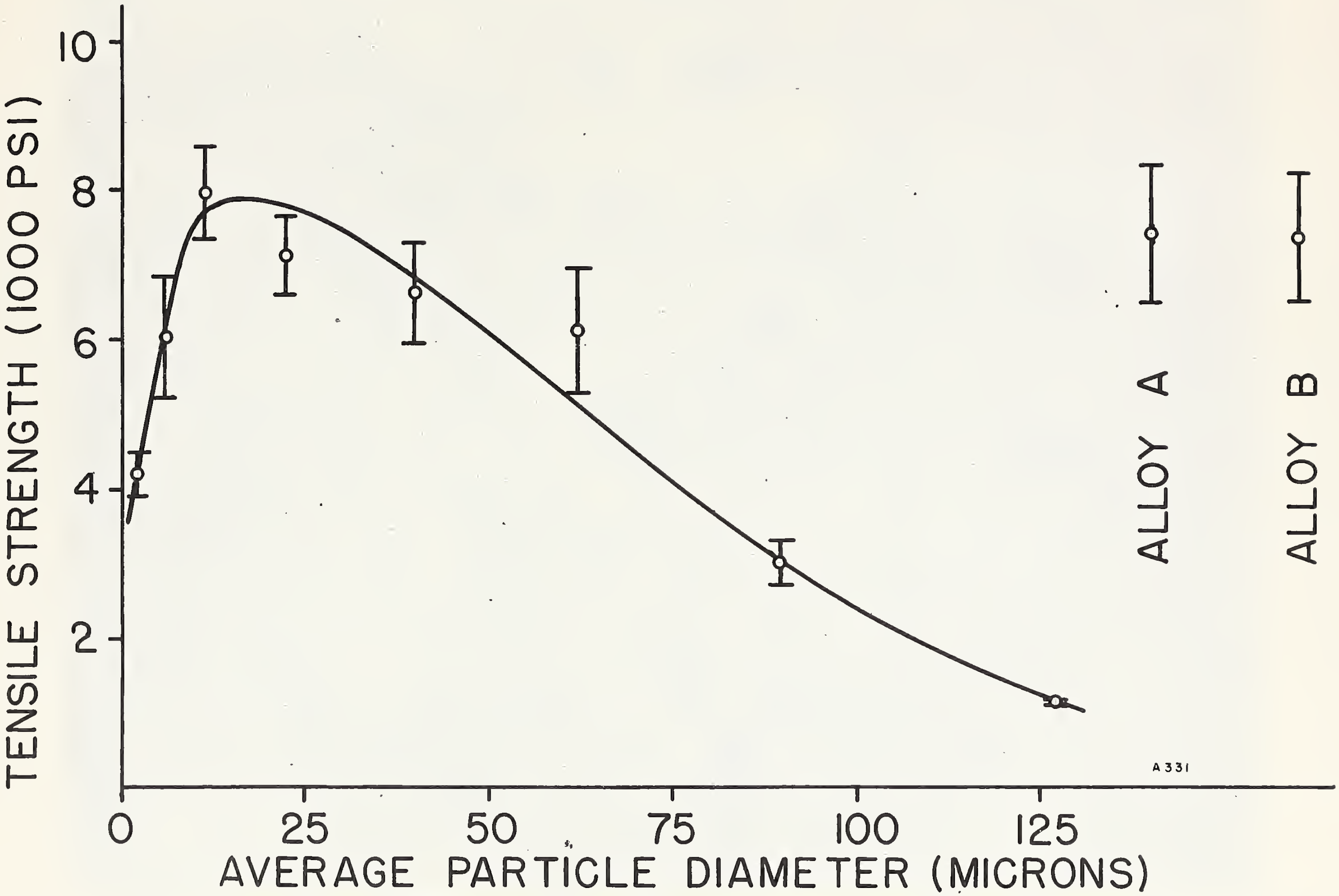

Figure 11. Tensile Strength vs Particle Size.

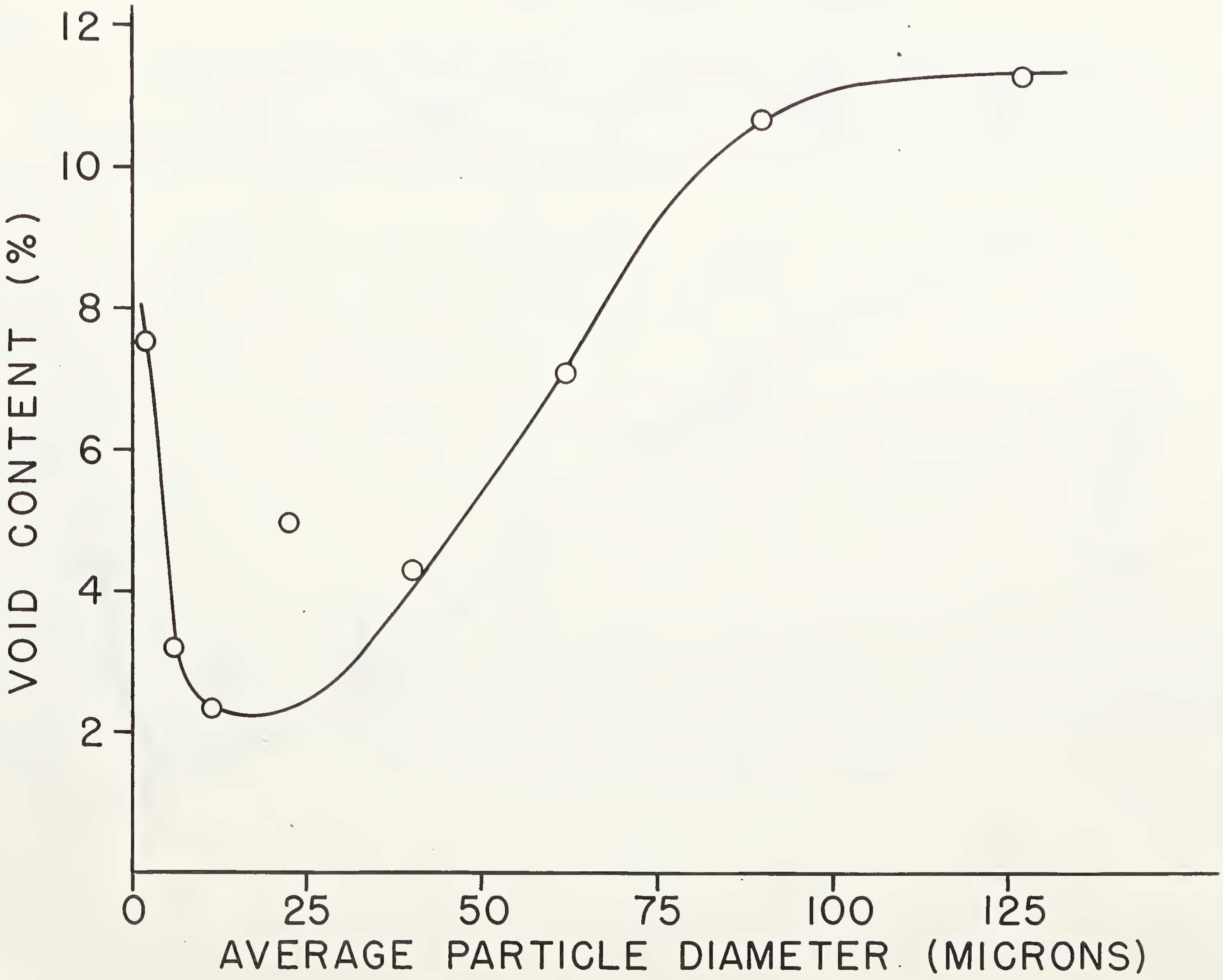

Figure 12. Void Content vs Particle Size

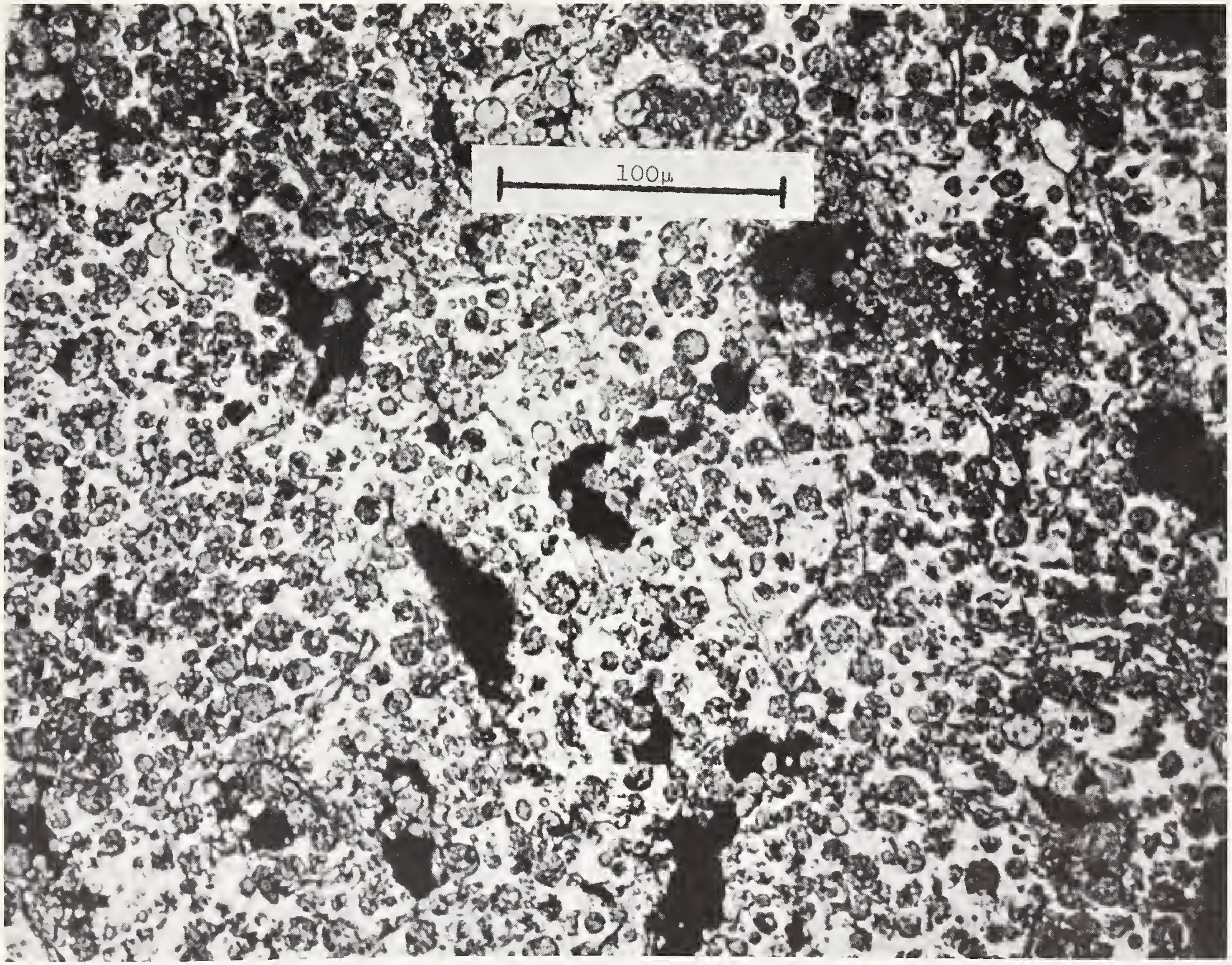

Figure 13-A. Spherical Alloy Amalgams Prepared at 1070 PSI Packing Pressure, 4-8μ Size Range.

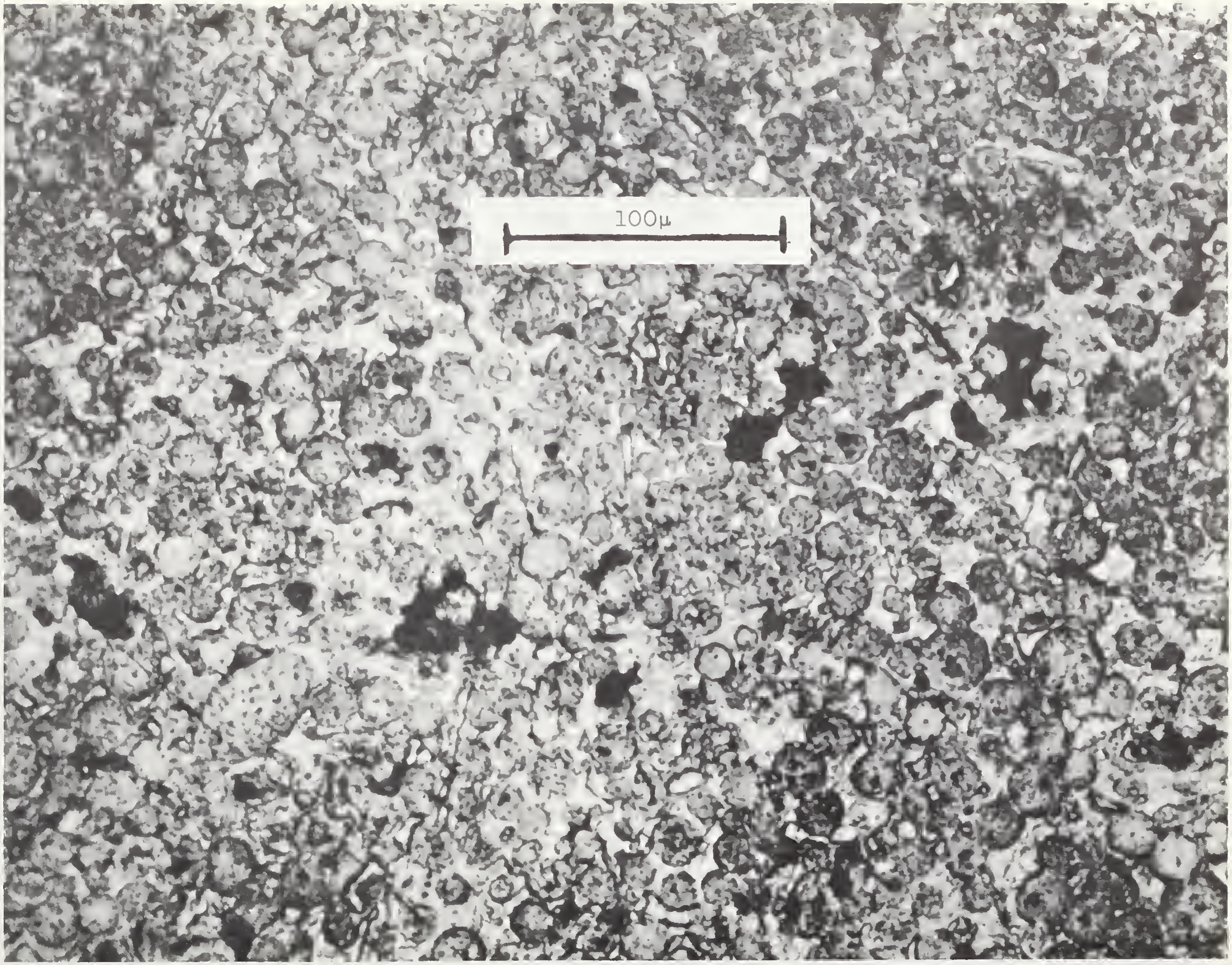

Figure 13-B. Spherical Alloy Amalgams Prepared at 1070 PSI Packing Pressure, 8-14μ Size Range.

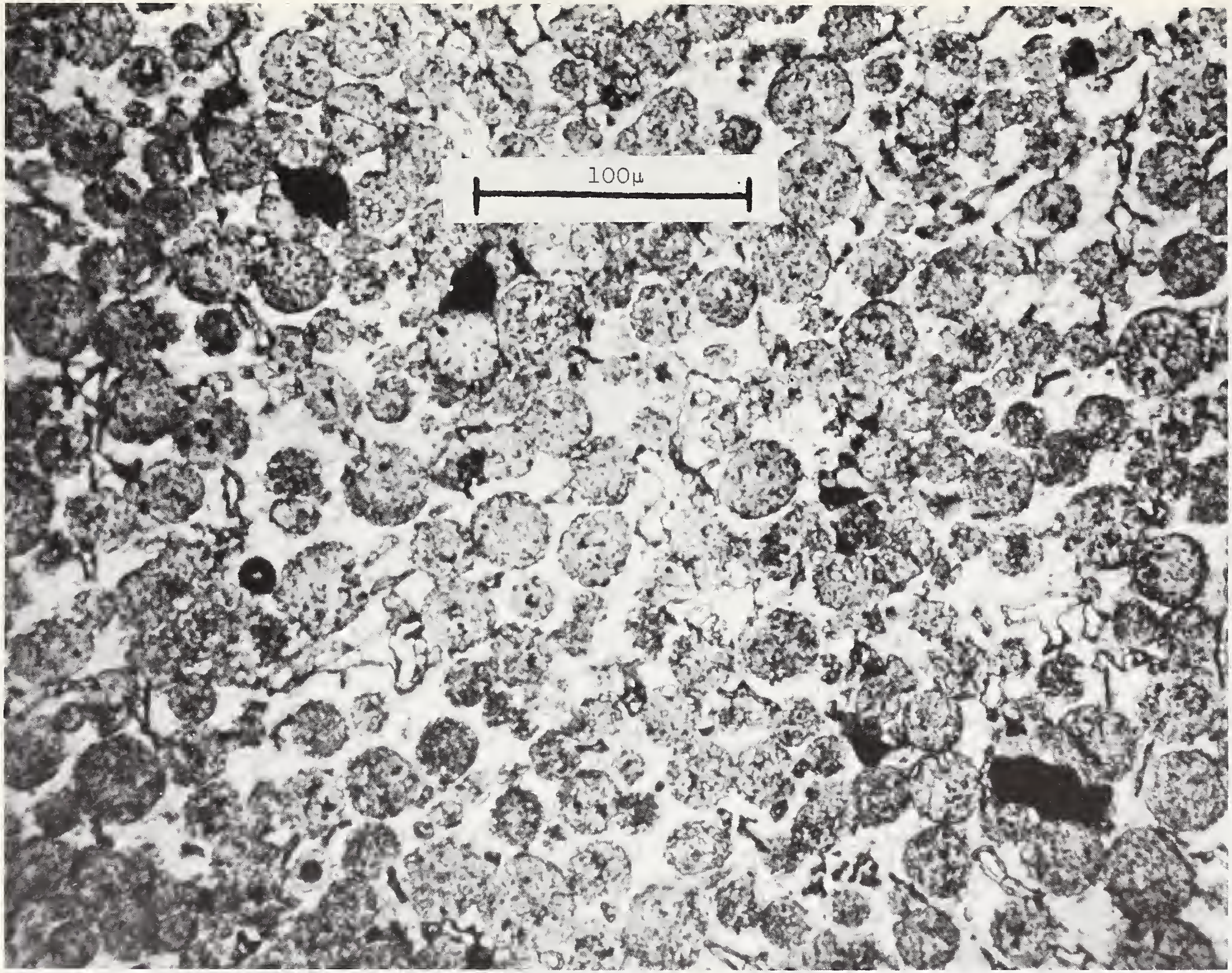

Figure 13-C. Spherical Alloy Amalgams Prepared at 1070 PSI Packing Pressure, 14-30µ Size Range.

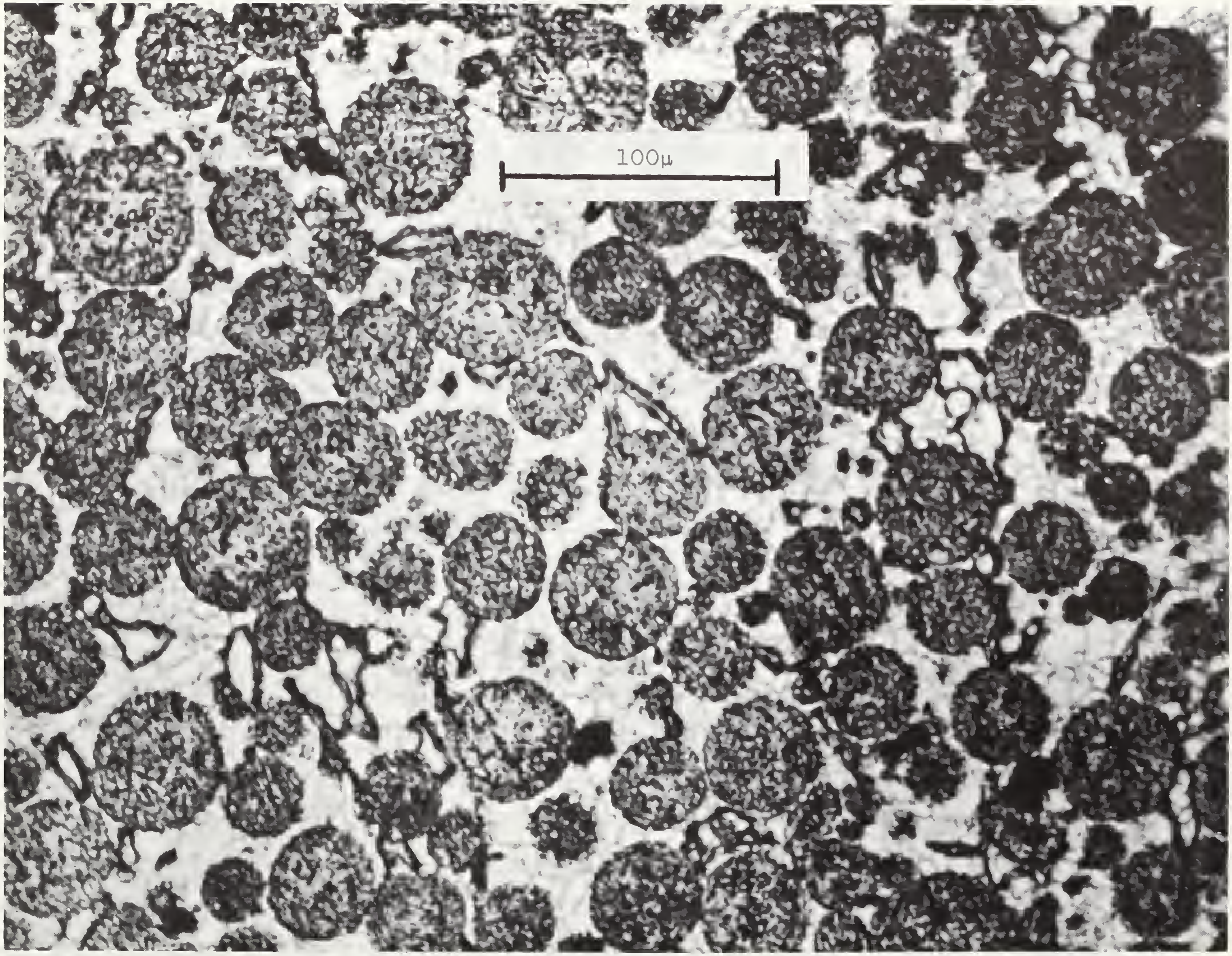

Figure 13-D. Spherical Alloy Amalgams Prepared at 1070 PSI Packing Pressure, 30-50µ Size Range.

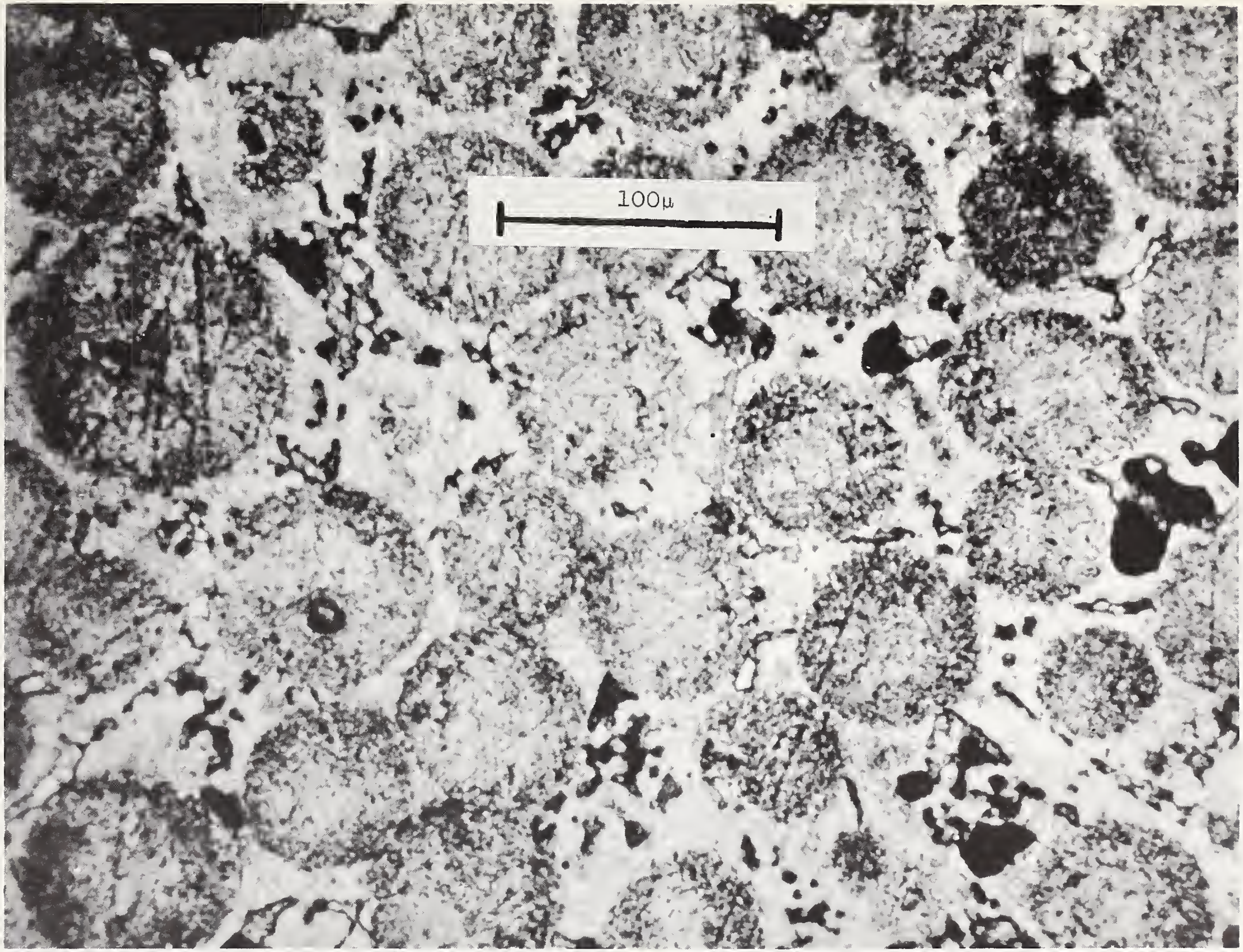

Figure 13-E. Spherical Alloy Amalgams Prepared at 1070 PSI Packing Pressure, 50-74μ Size Range.

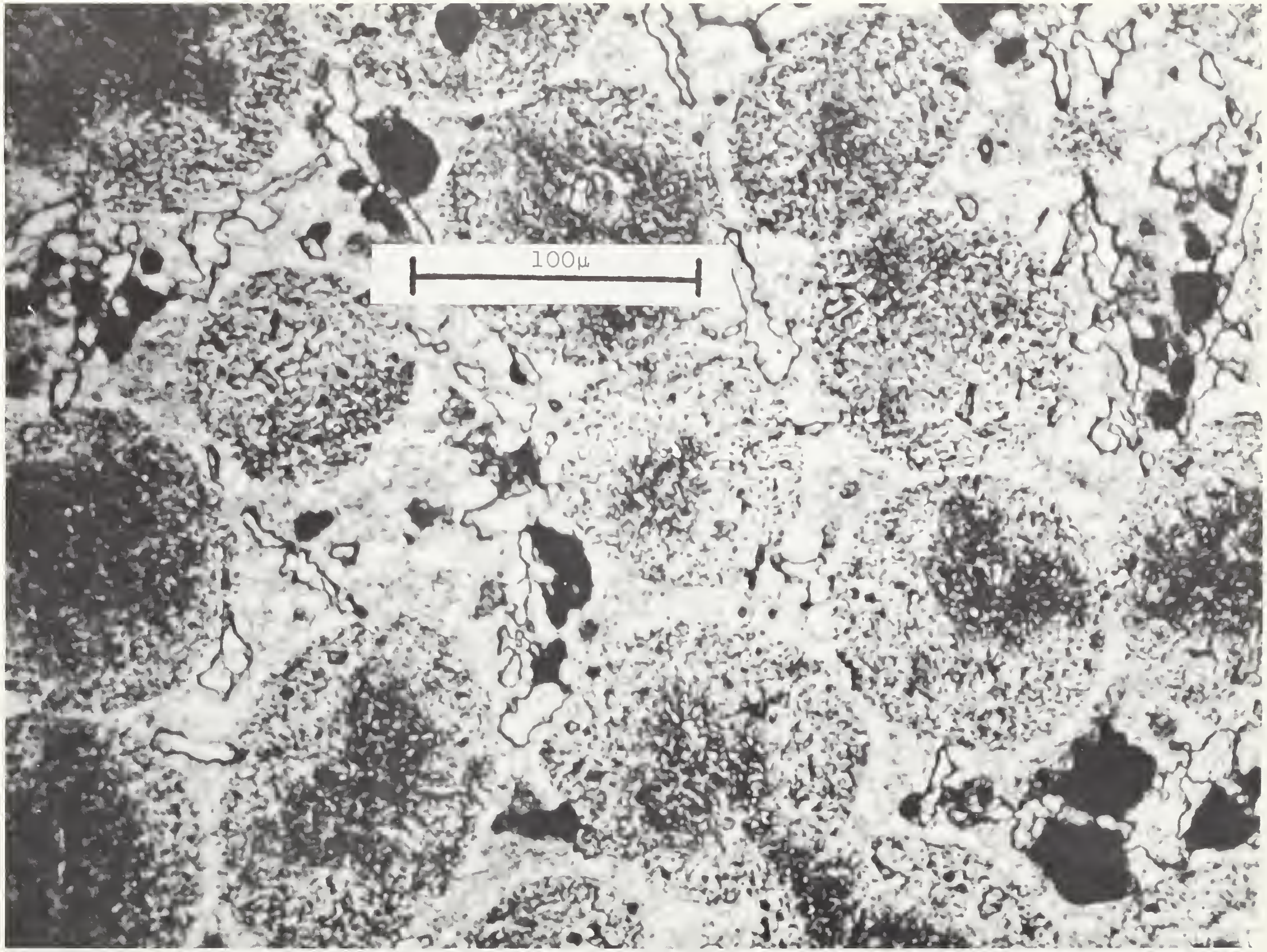

Figure 13-F. Spherical Alloy Amalgams Prepared at 1070 PSI Packing Pressure, 74-105µ Size Range.

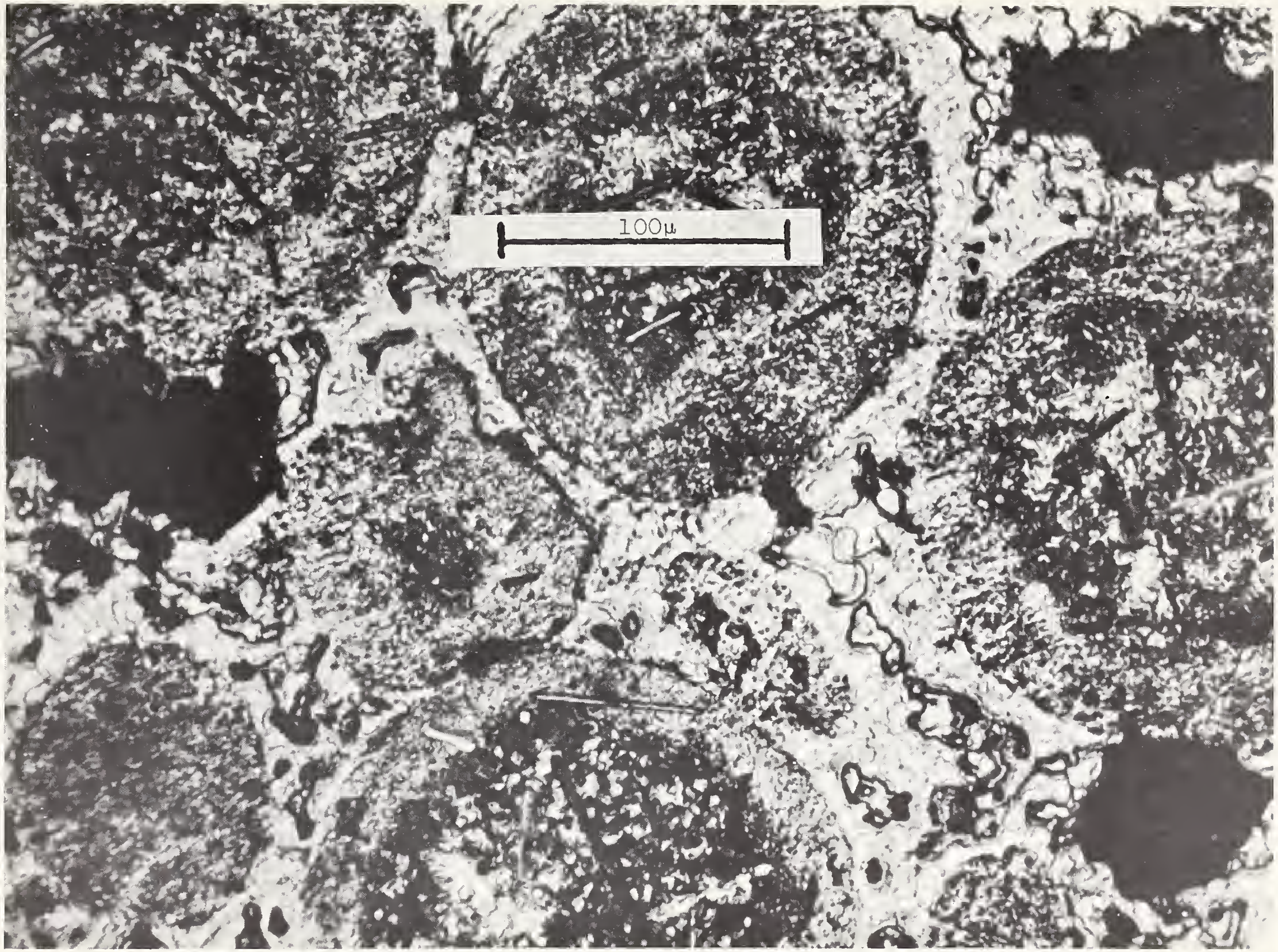

Figure 13-G. Spherical Alloy Amalgams Prepared at 1070 PSI Packing Pressure, 105-149μ Size Range.

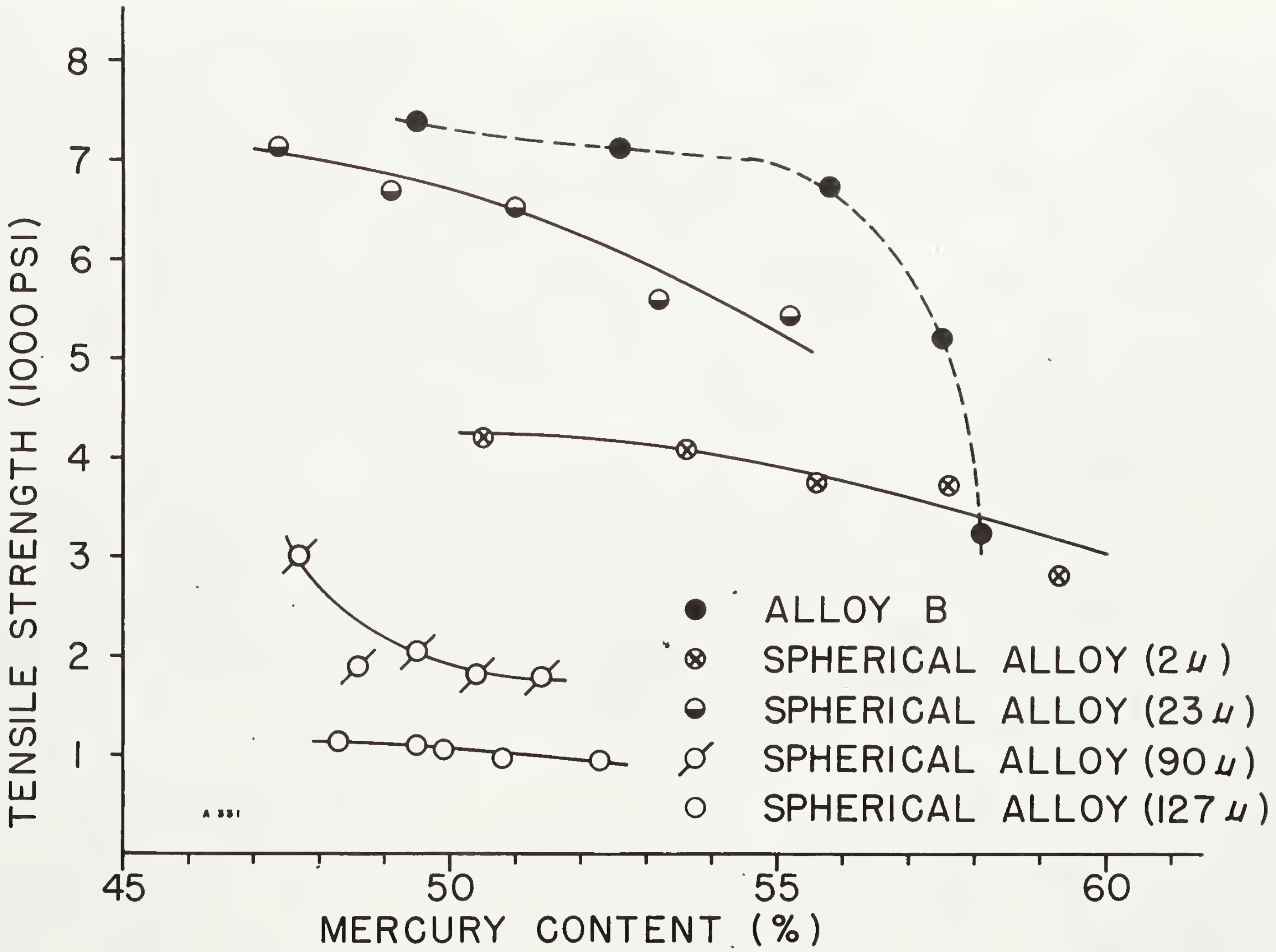

Figure 14. Mercury Content vs Tensile Strength. (Average particle sizes are given).

Figure 15. Adaptability of Spherical Amalgam (5) Compared with Conventional Amalgams (A and B) Packed at 95 PSI Pressure.