NATIONAL BUREAU OF STANDARDS REPORT

4313

Progress Report

on

ALLOYS OF GALLIUM WITH POWDERED METALS AS POSSIBLE REPLACEMENT FOR DENTAL AMALGAM

by

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U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

ALLOYS OF GALLIUM WITH POWDERED METALS AS POSSIBLE REPLACEMENT FOR DENTAL AMALGAM

Abstract

The possibility of using gallium as a liquid metal to replace mercury in dental restorations was investigated. It was found that gallium would not combine at room or mouth temperature with body-centered cubic metals such as tantalum, chromium and molybdenum. Gallium did combine with powdered face-centered cubic metals such as nickel, gold, copper, with alloys of copper and tin, and with cobalt.

With the exception of gallium-cobalt these alloys had adequate compressive strength and hardness but expanded excessively during hardening. Gallium-cobalt had low compressive strength but exhibited shrinkage during hardening.

Gallium alloys have been made that have some physical properties that are better than dental amalgam. None of the alloys investigated possess all the desired physical properties for oral restorations.

1. INTRODUCTION

The metallic element gallium was first predicted by Mendeleff in 1871 in his work on the correlation of the properties of the elements with their atomic weights [1]. It was also predicated at about the same time by de Boisbaudran who concluded, while studying the spectral lines of the elements, that there was an element missing between aluminum and indium in the periodic table. Gallium was first isolated by de Boisbaudran in 1875 [2].

The amount of gallium present in the earth's crust is about the same as lead, roughly 15 g per ton [3]. Unlike lead which is concentrated in localized areas, gallium is widely distributed. The low concentration of the ore and lack of demand for the metal make gallium relatively expensive at $2.50 to $5.00 per gram [3].

Gallium has several interesting physical properties. Its melting point is 29.78°C (85.60°F) [4] while its boiling point is 1983°C (3601°F) [4], thus giving it one of the longest liquid ranges of the metals. The metal has a low vapor pressure even at high temperatures, a pressure of 1 mm of mercury being attained at 1315°C (2399°F) [4]. Gallium expands 3.4% in volume during solidification, the solid having a density of 5.90 g/cm3. It has the property of wetting many materials including tooth structure.

The true nature of this property has not been determined but the wetting characteristic is generally attributed to a thin oxide film on the surface of the metal [6]. It has also been reported that gallium is essentially nontoxic [7].

This combination of physical properties indicates that gallium instead of mercury might be used as a component in a filling material. Some alloys of gallium begin to melt at higher temperatures than does dental amalgam. These factors make it desirable to investigate its possible use in oral restorations. It is expected that gallium, instead of mercury, might be used as a liquid metal to be combined with a powdered metal or alloy to form such a filling material. To be successful the combination should readily mix and condense, preferably using implements similar to those already familiar to the dentist. The gallium alloy should harden at mouth temperature within a reasonable length of time and possess strength, dimensional stability and corrosion resistance equal to or greater than those of dental amalgam.

2. MATERIALS AND METHODS

Gallium metal of 99.9+% purity was used in the experiments described here. The powdered metals and alloys used were obtained from laboratory supply houses, powdered metal manufacturers, or by alloying and comminuting in the Dental Research Section at the National Bureau of Standards. All metallic powders passed a No. 325 sieve of the U.S. Standard Sieve Series. For laboratory control, all mixing and condensing of specimens was at 37°C (98.6°F) which is above the melting point of gallium. Mixing was accomplished either in a mortar or in a mechanical amalgamator. All of the triturated mass was condensed. No liquid metal was expressed prior to or during condensation with one exception as noted.

Determinations of the dimensional change were made at 37°C (98.6°F) using unrestricted specimens 10 mm long and 5 mm in diameter in a differential interferometer with a fiducial reading 15 minutes after mixing was started. The compressive strength was determined at 21°C (69.8°F) using cylinders 8 mm long and 4 mm in diameter. The strength of one series of gallium-nickel-silicon alloys was determined at 37°C (98.6°F) and at 50°C (122°F). The rate of loading of the compressive strength specimens was in all cases 80 lbs (36.3 Kg) per minute.

3. RESULTS

Gallium-tantalum mixture. Two mixes of gallium and powdered tantalum were made, one containing 40% gallium and the other 25% gallium. The first was mixed with a mechanical amalgamator, the second with a mortar and pestle. Both specimens could be easily packed. These metals did not alloy. At room temperature the specimens hardened by the freezing of the gallium but readily softened when reheated to mouth temperature.

Gallium-chromium mixture. Gallium and finely divided chromium did not mix well. A specimen containing 35% gallium hardened by the solidification of the free gallium present. As in the case of the gallium-tantalum mixture, the gallium-chromium specimen softened by the melting of the gallium when heated to body temperature. A mix containing 50% gallium was too crumbly to pack.

When these mixtures of gallium and chromium were heated over an open flame the two metals combined with an exothermic reaction causing the mass suddenly to become bright red. The combination, after cooling, was a black friable powder.

Gallium-molybdenum mixture. Powdered molybdenum was mixed with 35, 40 and 50% gallium, respectively, in a mortar and in a mechanical amalgamator. The 50% gallium mixture was too wet to pack satisfactorily, while the 35% gallium mixture was too dry to pack well. The resultant mixtures did not harden during a 5-week period at room temperature. Small droplets of what appeared to be gallium were observed on the surface of the specimens.

Mixtures of gallium and molybdenum also combined exothermically when heated over an open flame to form a brittle, black mass.

Gallium-silver-indium alloys. Silver alloys containing 22½, 30 and 34% indium were prepared and chill cast into a steel mold. These alloys were then comminuted on a lathe. None of these alloys when combined with gallium hardened during storage at 37°C (98.6°F) for 1 month. The combinations remained soft and were easily deformed in the hand.

Gallium-gold alloy. A mixture containing 60% precipitated gold powder and 40% gallium hardened. These components react exothermically to form the intermetallic compound $AuGa_2$ [7]. The rate at which the reaction occurs is apparently dependent on the temperature at which the metals are mixed. Mixing temperatures of about 22°C (73°F) resulted in no appreciable heat generation; but when the components were mixed at a temperature of approximately 55°C (131°F) and packed into a 4 x 8 mm cylinder containing a thermocouple, temperatures as high as 450°C (842°F) were recorded.

The blue color, characteristic of $AuGa_2$ [7], was noted to extend throughout the specimen. These alloys had a linear expansion of about 12.5% during hardening.

Gallium-cobalt alloy. A mixture of 40% gallium with cobalt powder contracted approximately 3 μ/cm in 24 hours and was stable for 4 days thereafter. This is the only alloy investigated that contracted during hardening. The 7-day compressive strength of this alloy was 12,700 psi with a standard deviation of 1,100 psi. This low compressive strength makes the gallium-cobalt alloys unsuitable for oral use, but powdered cobalt may be introduced into an experimental gallium-powdered metal alloy to control an excessive expansion.

Gallium-nickel alloys. Nickel powder that passed a U.S. Standard Sieve No. 325 combined readily with gallium in several proportions. For practical purposes a gallium content from 20 to 40% was desirable. Less than 20% gallium resulted in a dry granular material that could not be readily packed and more than 40% gallium made a sloppy alloy that was difficult to handle. The data on compressive strength and hardness of four gallium-nickel alloys are summarized in Table 1. When triturated 70 revolutions in 30 seconds, the 35% gallium alloy expanded from 128 to 136 μ/cm at the end of 24 hours and was about constant at 140 to 149 μ/cm from 2 to 5 days (Fig. 1, Curves 1 and 2). Mixing this alloy in a mechanical amalgamator for 1 minute instead of a mortar for 30 seconds resulted in a 24-hour expansion of about 60 μ/cm (Fig. 1, Curve 3). This increased to about 75 μ/cm expansion after 3 days and remained constant for 14 days thereafter. Increasing the amount of work done on the mix appears to reduce the expansion and increase the compressive strength of the 35% gallium-nickel alloy.

Either powdered cobalt or powdered silicon reduced the large setting expansion of gallium-nickel alloys. For example, an alloy of 35% gallium, 55% nickel, and 10% cobalt expanded about 55 μ/cm at the end of 24 hours (Fig. 1, Curve 4). It continued to expand for about 6 days until a total expansion of approximately 70 μ/cm had occurred. No additional expansion had occurred at the end of 16 days. A specimen containing 36% gallium, 62% nickel and 2% silicon expanded 37 μ/cm in 24 hours. However, this method of reducing the high expansion of the gallium-nickel alloys also reduced their strength. A comparison of the 24-hour compressive strengths is given in Table 2.

The compressive strength of the 38% gallium, 60% nickel and 2% silicon alloy remained constant from 20°C (68°F) to 50°C (122°F). The strength of amalgam at mouth temperature was reduced approximately 21% and at 50°C (122°F) the strength was decreased about 43% (Table 3).

The tarnish resistance of gallium-nickel alloy was superior to amalgam when exposed to ammonium polysulfide vapor. The sulfide film that did form on the gallium-nickel alloy was easily removed by wiping with the finger, whereas the sulfide film on amalgam was adherent.

Gallium-copper and gallium-copper-tin alloys. Gallium combined with copper and copper-tin alloys of several compositions. Copper powder and a powdered alloy containing 90% copper and 10% tin were obtained from a manufacturer of powdered metals. Alloys with compositions corresponding to the γ (73% copper-27% tin), δ (68% copper-32% tin), ϵ (62% copper-38% tin) and η (41% copper-59% tin) phases of the copper-tin phase diagram (Fig. 2) [9] were made and comminuted. The compressive strengths of these,

when combined with 35% gallium, are shown in Figure 3. The alloy made by the combination of gallium and speculum metal (68% copper-32% tin) was readily mixed and packed and had good strength. This alloy hardened slowly as shown in the strength-time curve in Figure 4, and expanded from 46 to 50 μ/cm on hardening (Figure 5). This expansion is less than that of the 35% gallium-65% nickel alloy but is still high when compared to the expansion of dental amalgam (3 to 13 μ/cm is the range of expansion permitted by American Dental Association Specification No. 1 for Dental Amalgam Alloy) [10]. The hardness of this alloy varied from 100 to 119 Brinell which corresponds to the upper range of a type II (medium) or the lower range of a type III (hard) inlay casting gold alloy [10].

Gallium-copper-beryllium alloys. Gallium combined with a powdered copper-beryllium alloy (98% copper-2% beryllium) to form an alloy that hardened rapidly. The compressive strength (average of 2 specimens) of this copper-beryllium alloy when combined with 35% gallium was 19,500 psi at 1 hour and 18,500 psi at 24 hours. This would indicate that little or no reaction between the gallium and copper-beryllium alloy occurred after 1 hour. This does not agree with the data on dimensional change on hardening as shown in Figure 6. The technique for preparing the compressive strength specimens was the same as that for Curve 1, Figure 6. Different trituration times used in preparing this alloy resulted in different rates at which the expansion occurred (Figure 6). The discrepancy in the order of the expansion curves was not investigated because the alloy had unsatisfactory compressive strength.

When the gallium content was increased to 40%, liquid gallium squeezed out of a 1-hour-old specimen during crushing. This specimen had a compressive strength of 14,000 psi.

Gallium and commercial dental amalgam alloys (silver 65% min., tin 25% min., copper 6% max., and zinc 2% max.).* The combination of gallium with commercial dental amalgam alloys was unsatisfactory. The compressive strength of these alloys when mixed with gallium varied from a maximum of 14,000 psi after 1 week for a specimen containing 20% gallium, with a powdered alloy containing 69.1% silver, 26.8% tin, 2.8% copper and 1.4% zinc, to a minimum of 1,000 psi after 1 day when 35% gallium was triturated with an alloy containing 74.5% silver, 25% tin and 0.5% zinc. Trituration in all cases was in a mortar using 70 revolutions in 30 seconds with a 3 lb load on the pestle. The 1-day compressive strength of the 74.5% silver, 25% tin, 0.5% zinc alloys, when combined with gallium in this manner, decreased with an increasing gallium content as shown in Figure 7. The compressive strength of any of these is too low for use in the mouth.

* Composition limits for dental amalgam as per American Dental Association Specification No. 1 for Dental Amalgam Alloys (First revision 1934) [10].

4. DISCUSSION

This investigation indicates that powdered face-centered cubic metals such as gold, nickel, copper and some intermetallic compounds of copper and tin will, when triturated with gallium, harden at room or mouth temperature. Powdered body-centered cubic metals such as tantalum, chromium and molybdenum, when triturated with gallium, if they harden at all, do so too slowly to be of practical value.

The strength of alloys containing 35% gallium-65% copper, 35% gallium-65% speculum metal, 35% gallium-65% nickel is adequate for oral use but the expansion on hardening may be excessive as clinical experience with alloys having a setting expansion of about 50 μ/cm (35% gallium-65% speculum metal) has not been reported.

The wetting action of gallium should give good adaptation when the alloy is condensed in a cavity. Packing a 35% gallium-65% nickel alloy into a glass tube gave a mirror-like appearance to the inside of the tube. This wetting action is also a handicap when handling gallium alloys. The glass and metal mixing and condensing instruments become coated with gallium alloy making cleaning difficult. Since gallium does not wet wax and wax-like materials and smooth surfaces of some resins such as Teflon and Nylon, it may be possible to make satisfactory mechanical amalgamator capsules and pluggers from these resins.

The indication that gallium alloys retain their strength at mouth temperature and above, whereas amalgam does not, may be significant. Failure of amalgam at the margins of a restoration may be due, in part, to the reduced strength of the amalgam at body temperature.

5. SUMMARY

The following alloys of gallium with powdered metals and alloys have been made and found to harden at room or body temperature: 40% gallium-60% gold, 35% gallium-65% nickel, 38% gallium-60% nickel-2% silicon, 35% gallium-55% nickel-10% cobalt, 35% gallium-65% copper, 40% gallium-60% cobalt; and 35% gallium with the following copper-tin alloys; 90% copper-10% tin, 73% copper-27% tin, 68% copper-32% tin (speculum metal), 62% copper-38% tin, and 41% copper-59% tin.

Mixtures of gallium with tantalum, chromium, molydenum and some commercial amalgam alloys would not harden or were too weak to be of practical value.

The 40% gallium-gold alloy reacted exothermically under certain conditions and was therefore impractical for oral restorations.

The 35% gallium-nickel alloy had good strength and hardness but expanded 140 to 149 μ/cm in 2 days. The expansion was reduced to about 75 μ/cm by increasing the amount of work done on the mix during trituration.

Cobalt powder or silicon powder added to gallium-nickel alloys also reduced the expansion but the 1-day strength of the alloy was decreased by this treatment.

The 35% gallium-speculum metal alloy had good strength and hardness but set slowly. It continued to gain strength during 10 days storage at body temperature. This alloy had a setting expansion of 46 to 50 μ/cm.

Gallium alloys have been made with some physical properties that are adequate for oral restorations. However, sufficient data on the physical properties of gallium-powdered metal alloys are not available to recommend any so far investigated as possible replacements for dental amalgam. Prior to any such recommendations the alloy or alloys must be assessed biologically.

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TABLE 1

Compressive Strength and Brinell Hardness of Some Gallium-nickel Alloys

Composition Gallium Wt %	Composition Nickel Wt %	Compressive Strength psi	Baby Brinell hardness 15 Kg load 1/16" ball	Remarks
23	77	14,000	48	Triturated 2 min. 3 lb load on pestle. Specimen 3 days old
33	67	32,000	90	Triturated 2 min. 3 lb load on pestle. Specimen 1 day old.
35	65	49,000	90	Triturated 1½ min. 3 lb load on pestle. Specimen 1 day old
44	56	33,000	70	Triturated 2 min. 3 lb load on pestle. Specimen 1 day old
		62,000	100	As above except excess gallium was expressed during condensation.

Above values are individual or average of two determinations.

TABLE 2

Effects of Addition of Cobalt and Silicon to the Compressive Strength of Gallium-nickel Alloys

Composition Wt %	Compressive Strength Average psi	Standard Deviation psi
35 gallium 65 nickel	39,600	5,500
35 gallium 58½ nickel 6½ cobalt	31,100	2,300
38 gallium 60 nickel 2 silicon	25,500	800

All specimens made by triuration in a mortar, 70 revolutions in 30 seconds under a 3 lb load.

TABLE 3

Compressive strength (1 day) of amalgam and an alloy of 38% gallium, 60% nickel, and 2% silicon at 21°C, 37°C and 50°C (70°, 99° and 122°F)

Temperature	Amalgam*	38% gallium, 60% nickel, 2% silicon	
		Average	Standard Deviation
°C	psi	psi	psi
21	49,500**	25,500	800
37	39,000**	26,700	1,300
50	28,600**	25,200	2,000

* Alloy composition - Silver 70.3%, Copper 2.4%, Tin 25.5% and Zinc 1.8%

** Average of two determinations

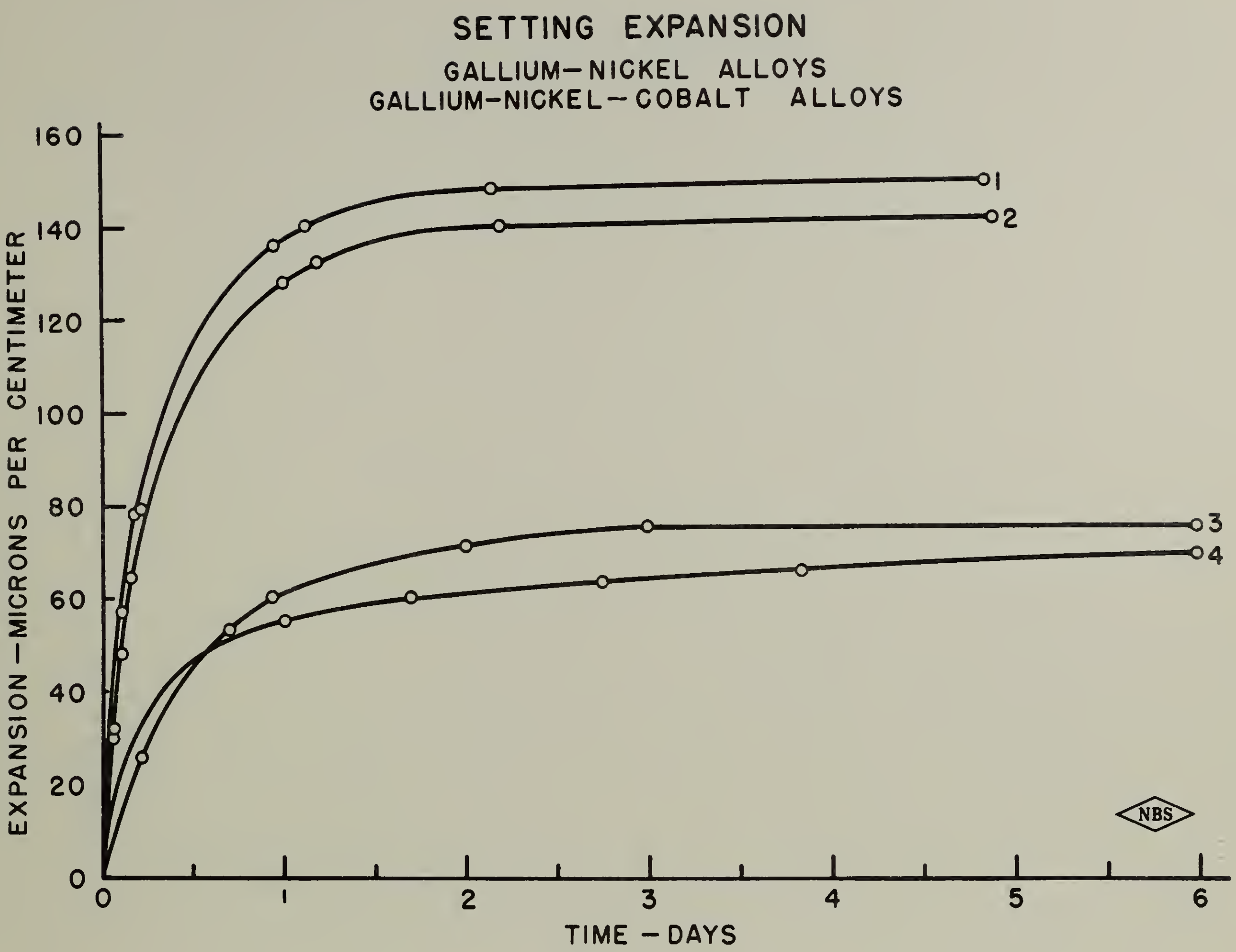
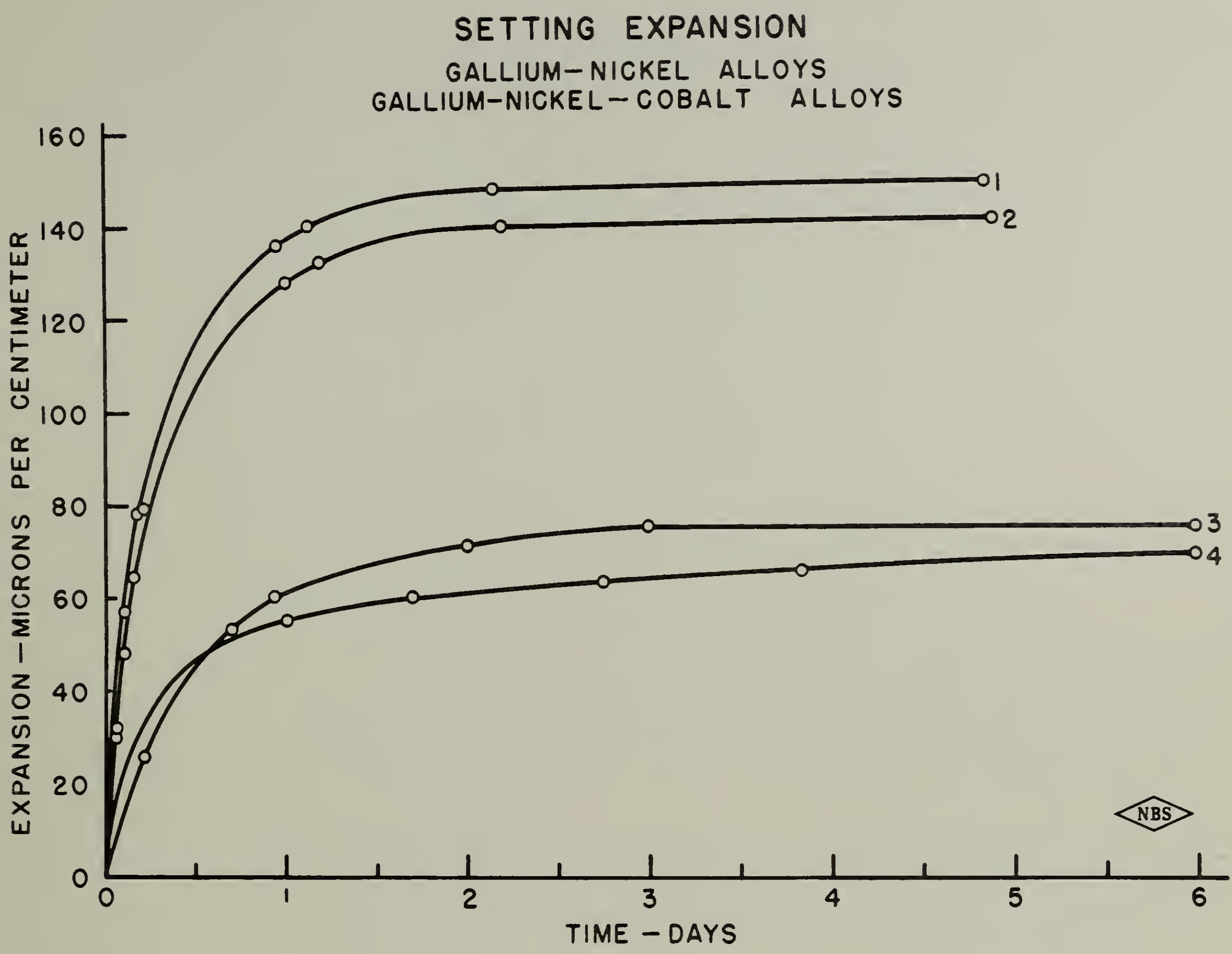


Figure 1. Dimensional Change on Hardening of Gallium-Nickel and Gallium-Nickel-Cobalt Alloys at 37°C (98.6°F)

Curves 1 and 2. 35% gallium-65% nickel triturated 70 revolutions in 30 seconds at 3 lb load.

Curve 3. Same as 1 and 2 except triturated 1 minute in a mechanical amalgamator.

Curve 4. 35% gallium-55% nickel-10% cobalt triturated 75 revolutions in 30 seconds at 3 lb load.



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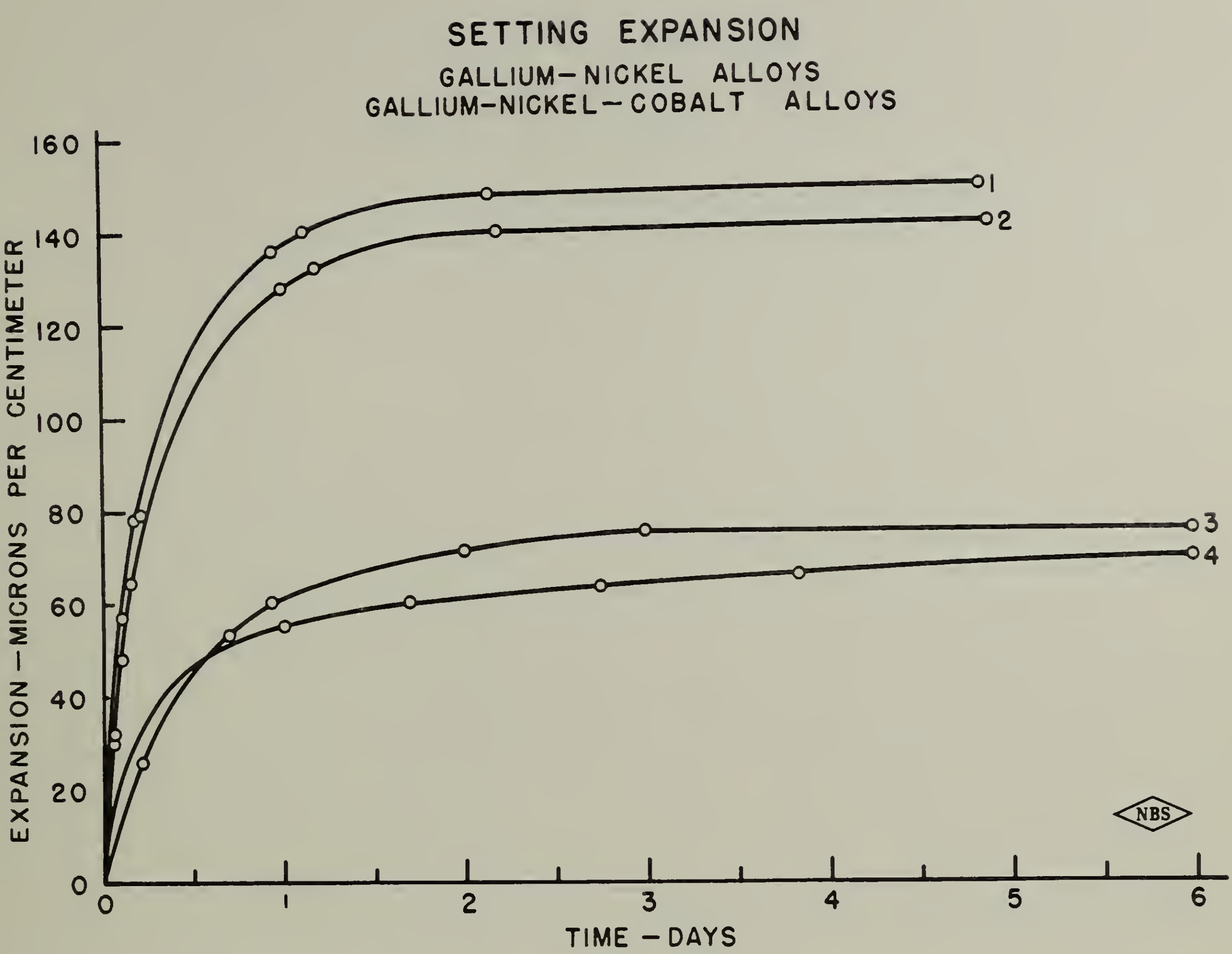

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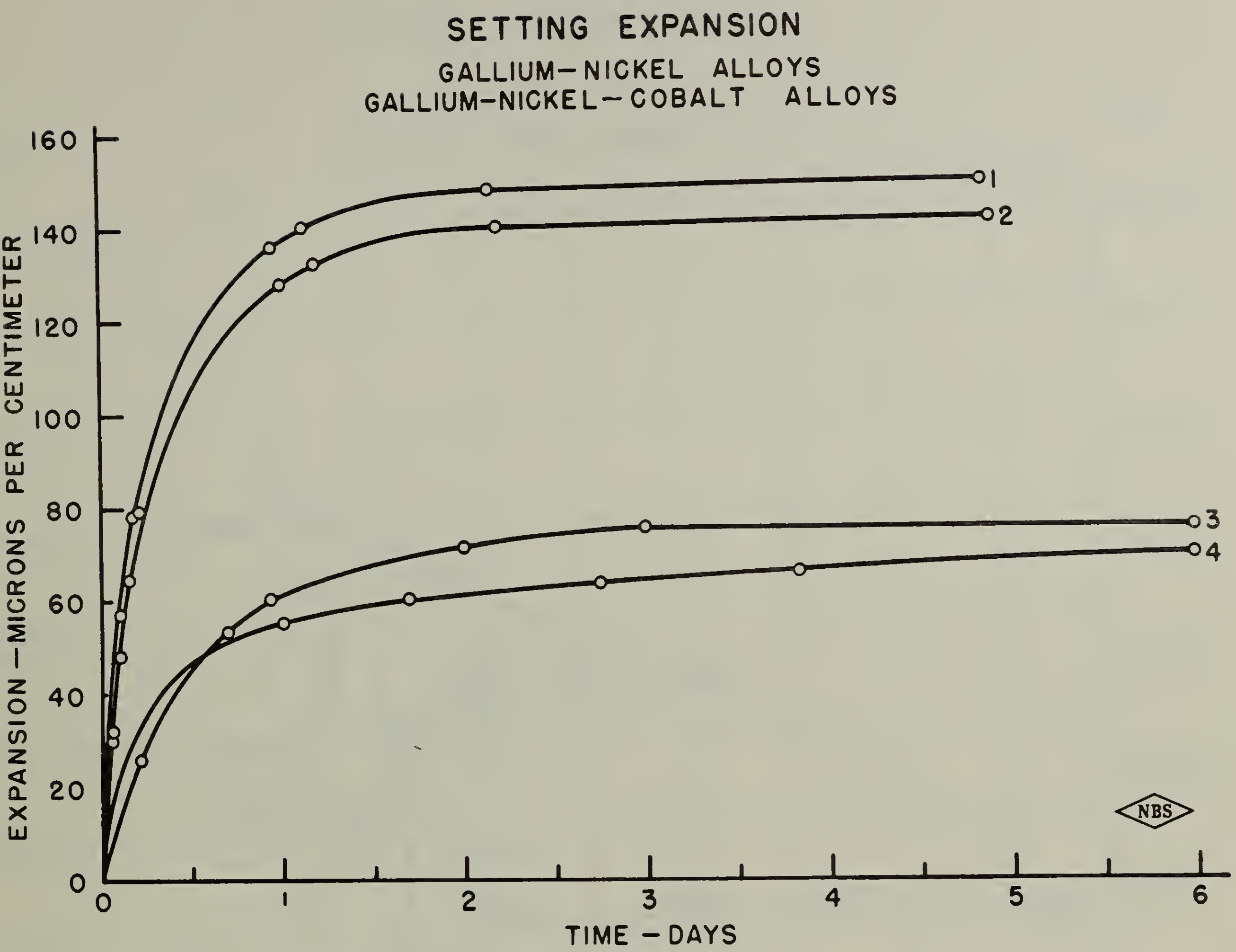

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Cu-Sn Copper-Tin

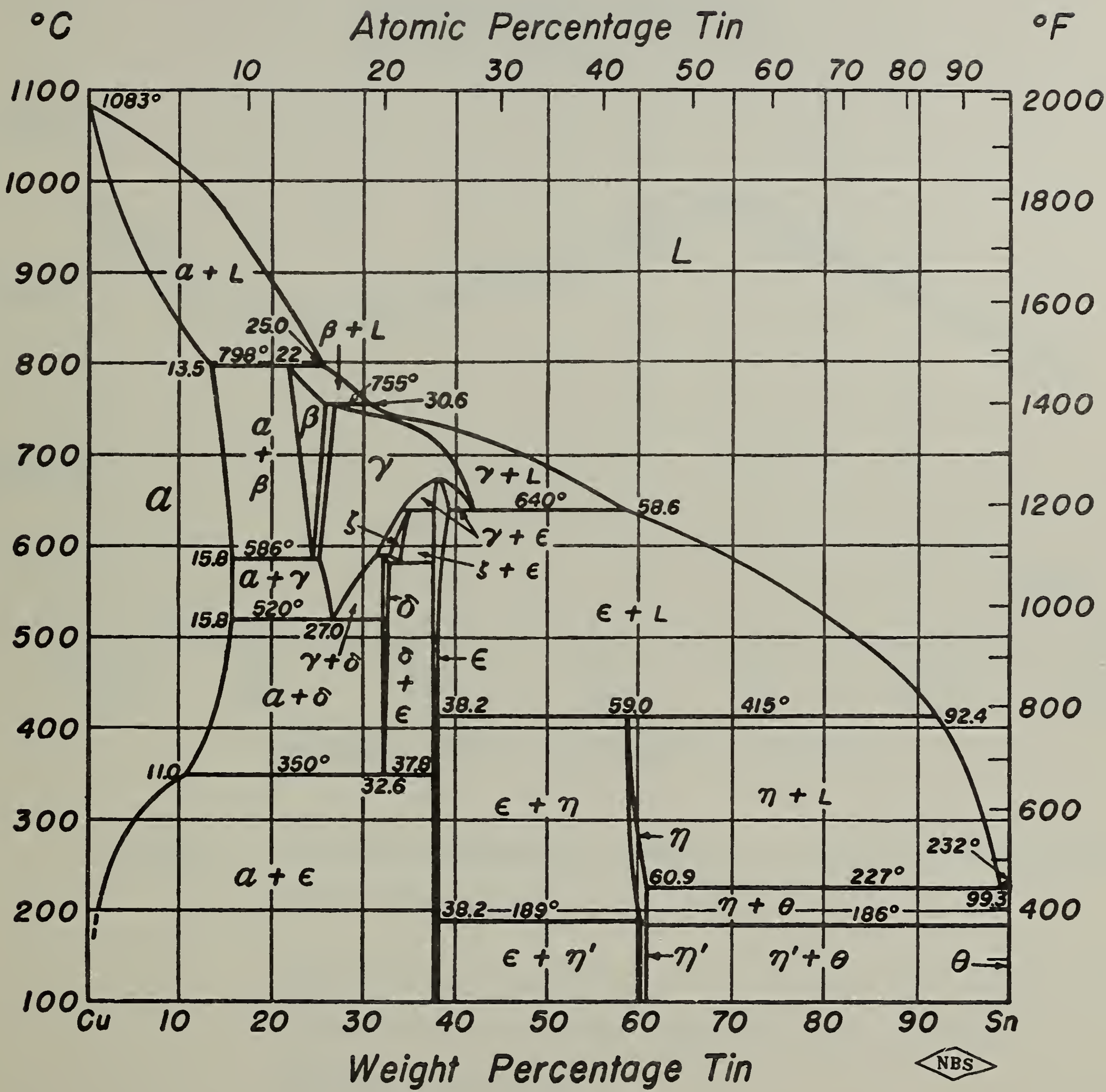

Figure 2. Copper-tin Constitution Diagram[9].

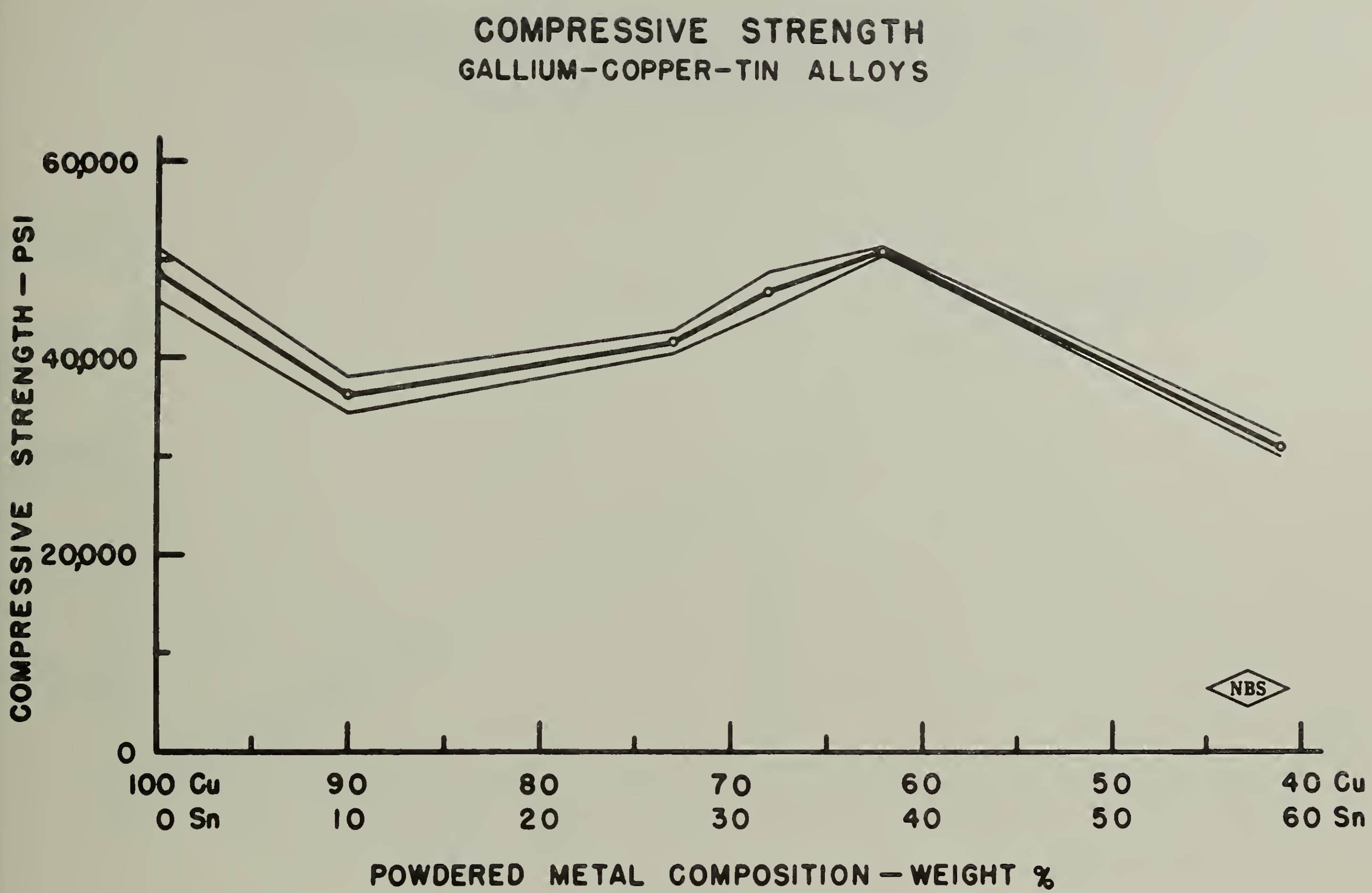

Figure 3. Three-day Compressive Strength of Copper and Copper-Tin Alloys when Combined with 35% Gallium.

Trituration in a mortar, 70 revolutions with a 3 lb load. Center line is average; upper and lower lines are standard deviations.

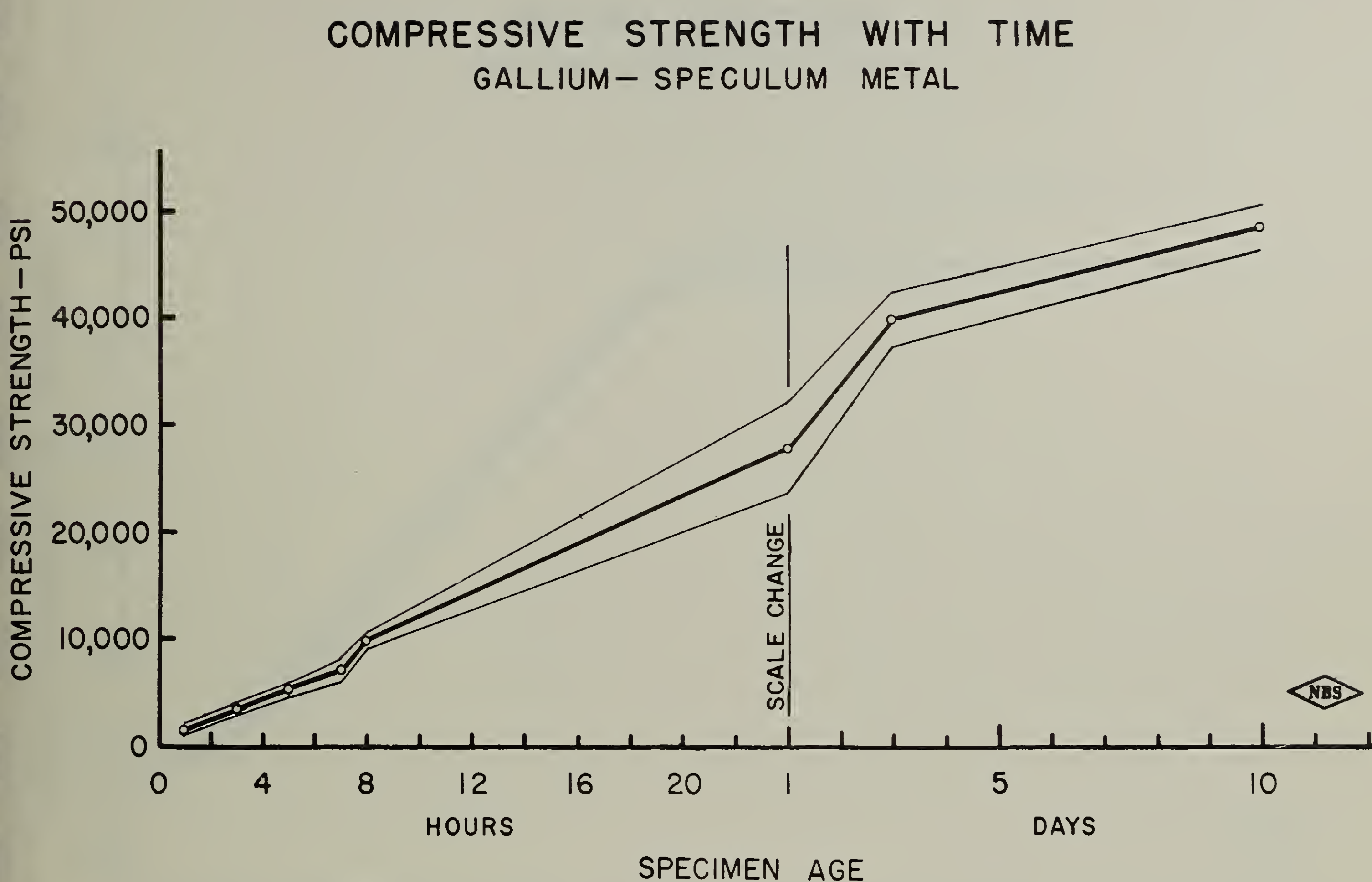

Figure 4. Compressive Strength of 35% Gallium, 65% Speculum Metal (68% copper-32% tin).

Triturated 70 revolutions in 45 seconds with a 3 lb load. Center line is average; upper and lower lines are standard deviations.

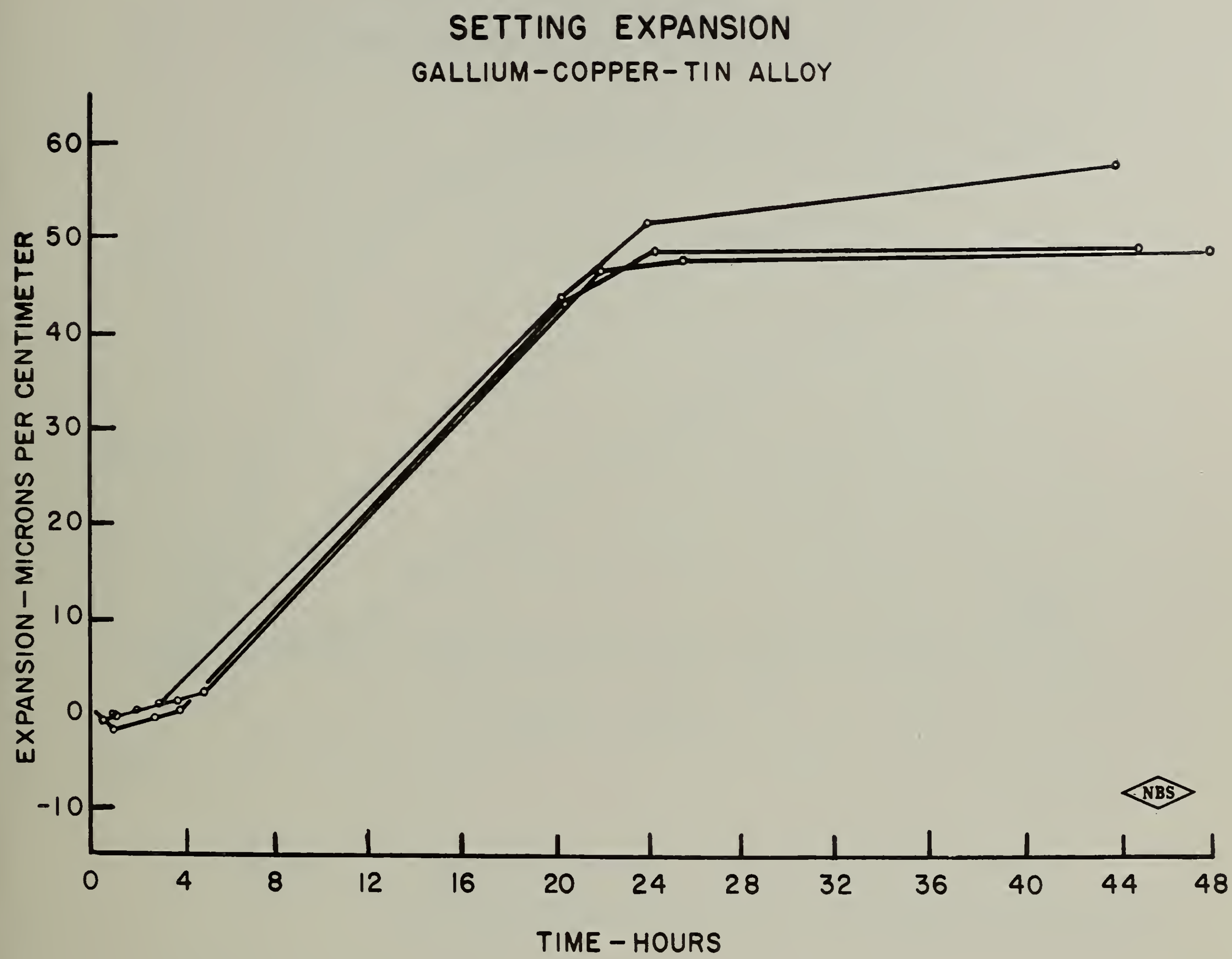

Figure 5. Dimensional Change on Hardening of Three Specimens of 35% Gallium-65% Speculum Metal (68% copper-32% tin).

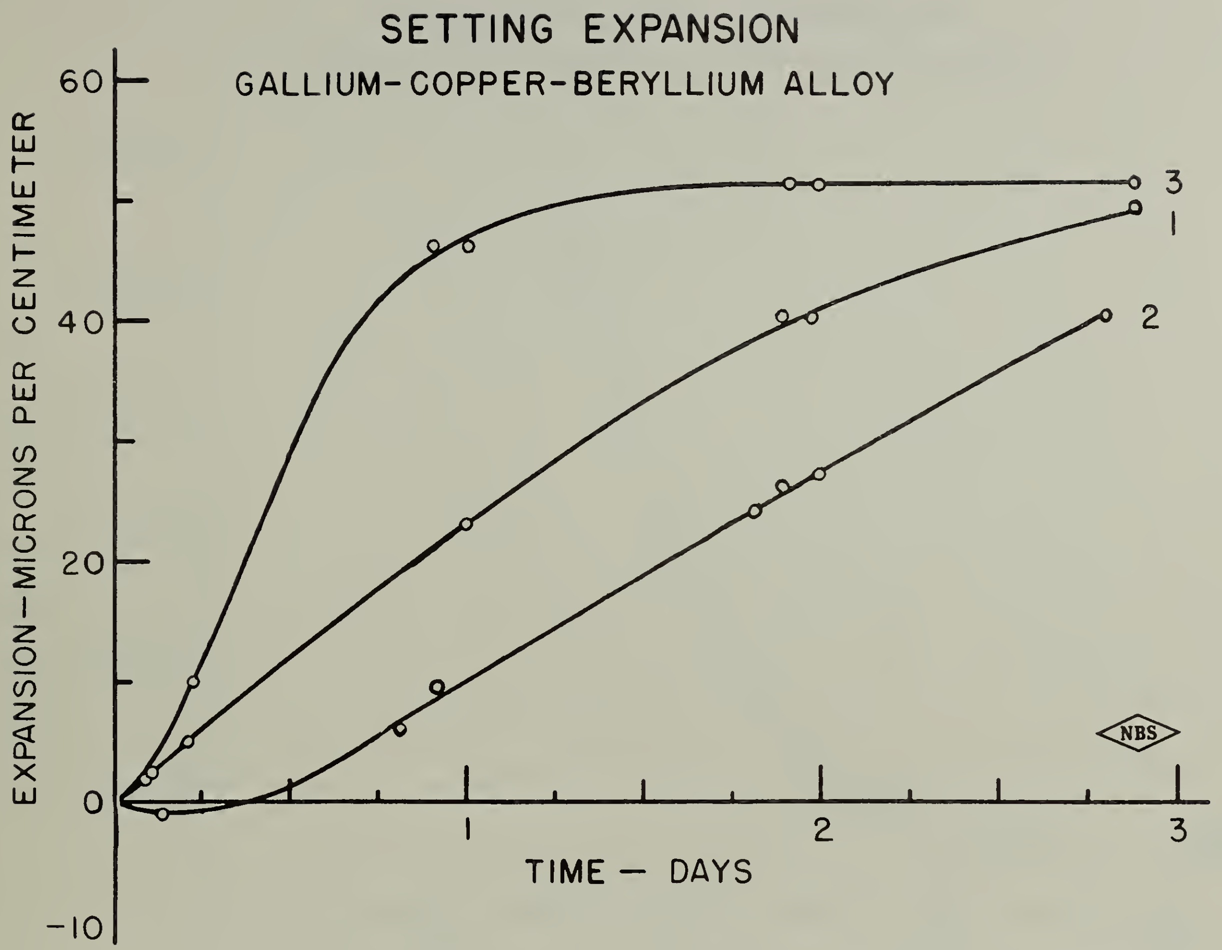

Figure 6. Dimensional Change on Hardening 35% Gallium-65% Copper-Beryllium Alloy (98% copper-2% beryllium).

Trituration in a mortar with 3 lb load.

Curve 1 - 50 revolutions in 30 seconds.

Curve 2 - 70 revolutions in 45 seconds.

Curve 3 - 100 revolutions in 60 seconds.

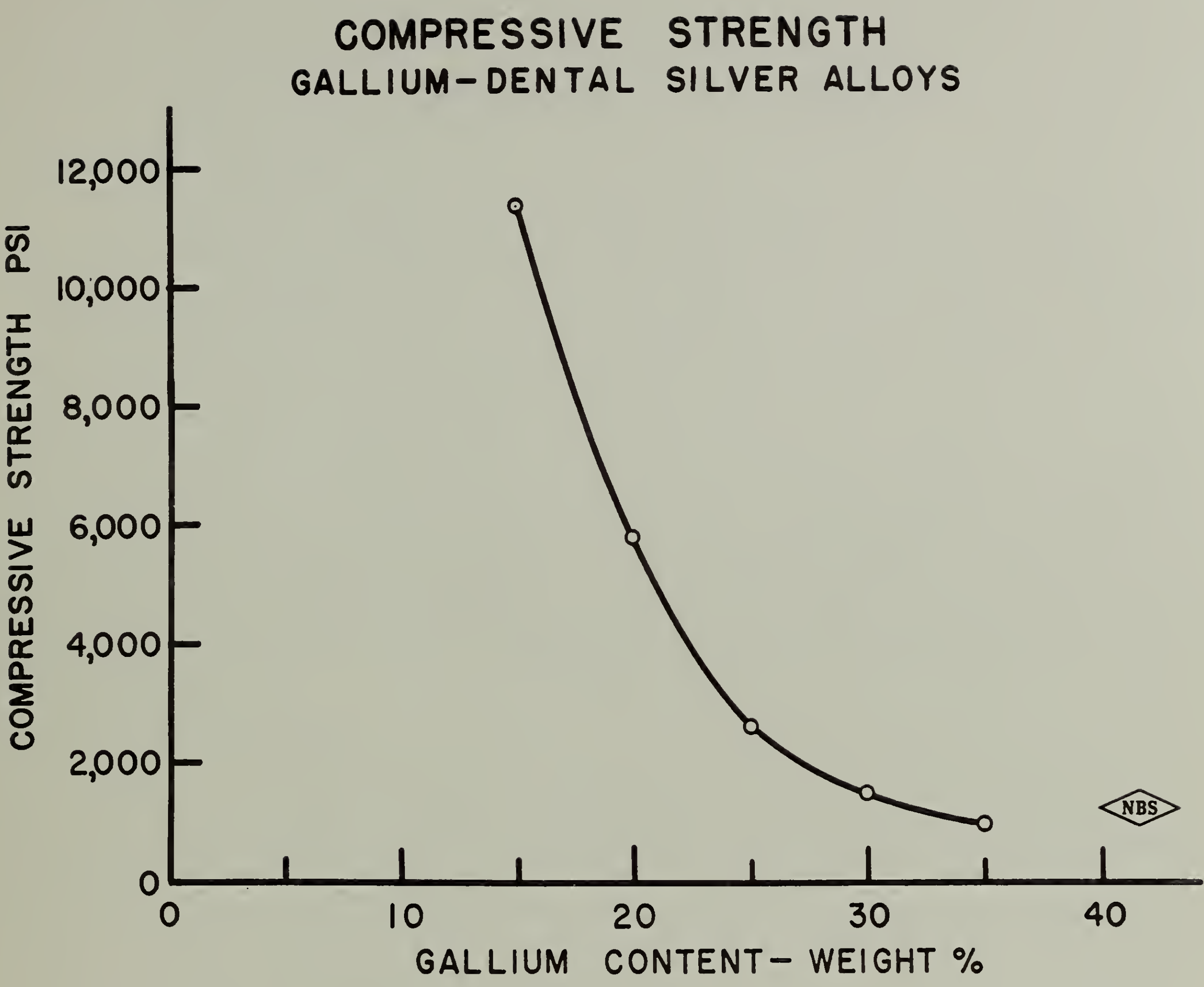

Figure 7. Compressive Strength at 24 hours of Gallium-Silver-Tin-Zinc Mixtures (powdered alloy composition, silver 74.5%, tin 25%, zinc 0.5%).
Triturated 70 revolutions in 30 seconds with 3 lb load.

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Reports and Publications

The results of the Bureau's work take the form of either actual equipment and devices or published papers and reports. Reports are issued to the sponsoring agency of a particular project or program. Published papers appear either in the Bureau's own series of publications or in the journals of professional and scientific societies. The Bureau itself publishes three monthly periodicals, available from the Government Printing Office: The Journal of Research, which presents complete papers reporting technical investigations; the Technical News Bulletin, which presents summary and preliminary reports on work in progress; and Basic Radio Propagation Predictions, which provides data for determining the best frequencies to use for radio communications throughout the world. There are also five series of nonperiodical publications: The Applied Mathematics Series, Circulars, Handbooks, Building Materials and Structures Reports, and Miscellaneous Publications.

Information on the Bureau's publications can be found in NBS Circular 460, Publications of the National Bureau of Standards ($1.25) and its Supplement ($0.75), available from the Superintendent of Documents, Government Printing Office. Inquiries regarding the Bureau's reports and publications should be addressed to the Office of Scientific Publications, National Bureau of Standards, Washington 25, D. C.

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