NATIONAL BUREAU OF STANDARDS REPORT

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Progress Report

on

SOME FACTORS AFFECTING
THE DIMENSIONAL CHANGES
OF GOLD-ALLOY INVESTMENTS

by

Avelino A. Macasaet
George Dickson

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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Avelino A. Macasaet*
George Dickson**

* Guest Worker, Georgetown University
Dental Research Section, National Bureau of Standards

** Physicist, Dental Research Section,
National Bureau of Standards

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U. S. DEPARTMENT OF COMMERCE

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SOME FACTORS AFFECTING THE DIMENSIONAL CHANGES OF GOLD-ALLOY INVESTMENTS

Abstract

The effects of water-powder ratio, water addition and heating rate on the setting and thermal expansions of investments were studied. Increasing the water-powder ratio decreased the thermal expansion. The addition of water to investments during setting increased the setting (or hygroscopic) expansion and reduced the thermal expansion. Variation of heating rate changed the shape of the thermal expansion curve but generally had little effect on the thermal expansion at 700°C.

1. INTRODUCTION

Studies have shown that the setting, hygroscopic and thermal expansion properties of dental gold-alloy investment materials are affected by a number of variables, [1-6]. Asgar, Mahler and Peyton [7] showed that the hygroscopic expansion of an investment material is related to the amount of water added following a given mix. An almost linear relationship was demonstrated between the amount of water added and the resulting hygroscopic expansion. A method of compensation for the casting shrinkage of gold alloys based on the principle of controlled water addition was developed.

Hollenback and Rhoads [8] found that an increased hygroscopic expansion was accompanied by a decrease in thermal expansion. With increased hygroscopic expansion, the combined total expansion was also increased. They reported, however, that when the total linear expansion of the mold cavity was measured during setting either in air or in water and during heating to 700 or 800°C the resulting total expansion was approximately 1.40%. This value was in agreement with Hollenback and Skinner's [9] reported casting shrinkage for gold alloys of from 1.29 to 1.56%.

The present study was undertaken to obtain further information on the relationships between setting, hygroscopic and thermal expansion and to determine the effects of water-powder ratio and heating rate on the dimensional changes of investments.

2. MATERIALS AND METHODS

The investment materials used in this study are listed in Table 1. Their compositions as determined by petrographic examination are given in Table 2. The thermal expansion technic is usually employed with materials A and B while the hygroscopic technic of compensation is used with materials C and D.

In order to obtain information on the total dimensional change of the investment during setting (either with or without added water) and during heating, the same specimen was generally used for both setting and thermal expansion measurements. This required the use of a specimen with small cross section so that it could be inserted into the fused quartz apparatus for thermal expansion determinations.

To prepare the specimens, 40 grams of the investment material was weighed out using a torsion balance accurate to the nearest 0.01 gm. The weighed investment was placed in a plaster bowl which was damp dry. A graduated cylinder calibrated in ml. was used to measure the required amount of distilled water for a particular mix. Water was measured to the nearest 0.2 ml. Water-powder ratios were varied from 30/100 to 40/100.

Hand spatulation was employed for thirty seconds in mixing the investment with water. About 50 turns of a plaster spatula were made to produce the homogeneous mix. This was followed by thirty seconds' vibration of the mix on a mechanical vibrator, to remove as much as possible any air bubbles which may have been entrapped in the mix.

Specimens for setting expansion were formed and measured in U-shaped molds 10 inches long and 11/16 of an inch wide, lined with rubber dam. A micrometer microscope comparator and V-trough were utilized to measure the setting (and hygroscopic) expansion. The initial reading was taken immediately after the specimens were placed in the trough under the microscope. The final reading was taken two hours after the start of mix. To produce hygroscopic expansion a specific amount of distilled water was added to the top of the specimen 3 minutes after start of mix using a tuberculin syringe graduated in 0.01 cc.

Thermal expansion was determined for investment specimens heated from room temperature to 700°C at uniform heating rates of 3.75, 5.62 and 11.25°C per minute. Measurements were also made on specimens which were held for one hour at constant temperatures of 330 or 380°C during the heating cycle.

Thermal expansion was determined with a fused quartz tube apparatus and dial guage. The specimen for setting expansion was cut to 4 inches for the thermal expansion test with the junction of a 28 gauge chromel-alumel thermocouple (embedded when the specimen was prepared) at the center of the 4 inch specimen.

The specimens which were used for fast heating and maintaining at 330°C and 380°C for one hour were prepared in split brass molds 8 inches long with a diameter of 1/2 inch. The junction of a chromel-alumel thermocouple was also embedded in the center of the 8 inch specimen. The specimen was heated to 330 or 380°C and held at this temperature for one hour. Then a heating rate of 3.75°C per minute was followed until a peak temperature of 700°C was reached.

3. RESULTS

3.1 Effect of Water-Powder Ratio on Setting Expansion

Variation of water-powder ratio within the range studied, 30/100 to 40/100, did not seem to have an effect on the setting expansion of investment A. Specimens with water-powder ratios of 30/100 and 40/100 had average setting expansions of 0.36 and 0.34% respectively as shown in Table 3. Increase in water-powder ratio tended to decrease the setting expansion of investment C and D both in air and in water, Table 4. However, over the range investigated this decrease was not large compared to the variations of individual specimens.

The addition of increasing amounts of water to investment specimens three minutes after the start of mixing produced corresponding increases in hygroscopic setting expansion. This is shown in Figure 1 and Table 5 for investments A and B. The vertical bars on Figure 1 represents the range in hygroscopic expansion of three specimens.

3.2 Effect of Water-Powder Ratio on Thermal Expansion

The effects on thermal expansion of variations in the water-powder ratio of a cristobalite and a quartz-containing investment are shown in Figures 2 and 3. In Figure 4 is shown a comparison of the thermal expansions of two series of specimens of investment A with water-powder ratios varying from 30/100 to 40/100. In one series the investments were mixed with different amounts of water to produce the desired ratios. In the other series all specimens were mixed with a ratio of 30/100, and different amounts of water were added later to produce hygroscopic expansion and bring the water-powder ratios up to the desired values. The lines passing through the points in the graph represent the average thermal expansion at 700°C of two or more expansion runs made on each mix. The vertical bars show the ranges in thermal expansions. It is evident that an increase in water-powder ratio produced a corresponding decrease in thermal expansion in both series of specimens.

The similarity over the entire temperature range of thermal expansion curves for specimens with the same final water-powder ratios, whether all of the water is introduced in the initial mix or whether part is introduced initially and the remainder added later to produce hygroscopic expansion, is shown in Figures 5 and 6. Shown in each of the figures are two thermal expansion runs on specimens of investment A with the same final water-powder ratio. The only difference is the time at which part of the water was made available to the investment material. For Figures 5 and 6, curves with nearly identical expansion values at 700°C were selected to illustrate the similarity of the shapes of the curves. As shown in Figure 4, the normal variation of duplicate specimens at 700°C was greater than the variation of the pairs selected. Values for total expansion (setting or hygroscopic plus thermal) are given in Tables 3 and 5. Data in Table 3 indicate that for specimens not expanded hygroscopically, increasing the water-powder ratio produced a decrease in the total expansion. Table 5 indicates that for investments A and B, mixed with a water-powder ratio of 30/100, the addition of water in the amounts listed to produce hygroscopic expansion had little effect on the total expansion, since the increase in hygroscopic expansion was only slightly greater than the accompanying decrease in thermal expansion. The addition of water had a greater effect on the total expansion of investment A when the original water-powder ratio was less than 30/100 as shown in Table 6.

3.3 Effect of Heating Rates on Thermal Expansion

The use of different uniform heating rates varying from 3.75 to 11.25°C per minute did not produce any significant change in the thermal expansion at 700°C of investments A, B, C and D, Table 7 and Figures 7 and 8. A faster heating rate, 19°C per minute, did increase the thermal expansion of investment C at 700°C.

4. DISCUSSION

The results obtained on dimensional changes of investments during wetting, particularly the results on investment A, show that the normal setting expansion varies only to a slight extent with water-powder ratio while hygroscopic expansion increases significantly as the amount of water added is increased. These results are in agreement with those reported by other investigators and are of interest in this study primarily as they relate to thermal expansion of investments.

In general the data indicate that setting or hygroscopic expansion as such have little effect on thermal expansion but that the change in water content inherent in the hygroscopic technic is a significant factor in varying the thermal expansion.

The thermal expansion of investment was found to depend more upon the amount of water incorporated in the specimen than upon whether this water was in the initial mix or was added later to produce hygroscopic expansion. Although it is true that the addition of increasing amounts of water increased the hygroscopic expansion of investments, the combined hygroscopic and thermal expansion of the investment materials was not increased a like amount. This is shown in Tables 5 and 6.

The decrease in thermal expansion of specimens with increased water-powder ratio, whether the water was all introduced in the initial mix or whether some of it was introduced later to produce hygroscopic expansion, may be explained as follows. In both instances the additional water increases the volume of the specimen, reduces the ratio of refractory volume to specimen volume and increases the porosity of the specimen. With increased porosity there is greater opportunity for the refractory particles to expand into the porosities without increasing the overall dimensions of the specimen.

The results of the effect of different heating rates on thermal expansion indicate that variation of heating rates (11°C per minute or slower) does not have a significant effect on the thermal expansion at 700°C. Variation of heating rates however alters the thermal expansion curves at lower temperatures particularly in the 330 to 400°C range.

The usual thermal expansion curve of a gypsum investment material shows a contraction within the 300 to 400°C range. Actually the investment is undergoing two types of dimensional change at this time, a thermal expansion and a contraction resulting from loss of water of crystallization. When the investment is held at a constant temperature within this range, the shrinkage occurs at that temperature and subsequent heating results in expansion. Since the shrinkage due to loss of water takes place slowly, faster heating rates spread this shrinkage over a greater portion of the temperature range and thus alter the shape of the thermal expansion curve. At the heating rate of about 19°C per minute a similar shrinkage effect with the quartz-containing investment at about 100°C appears to be almost completely suppressed with a resulting higher expansion of 700°C. This was the only variation in heating rate which significantly affected the expansion of the investment at 700°C.

CONCLUSIONS

Increasing the water-powder ratio of investment reduces the thermal expansion. The addition of water to an investment material to produce a hygroscopic expansion generally produces a lower thermal expansion.

Where the proportion of water to investment is kept constant there are only small differences in the thermal expansion of investment specimens in which all of the water was added in the initial mix and specimens in which part of the water was added later to produce hygroscopic expansion.

The amount of water added to an investment material after the initial mix affects its hygroscopic expansion but has less effect on its total or combined expansion.

Heating rates of 11°C per minute or slower did not have a significant effect on thermal expansion of investment materials at 700°C. A heating rate of 19°C per minute produced a higher thermal expansion at 700°C in a quartz investment. The shape of the thermal expansion curve of investment, particularly in the 330 to 450°C range, was dependent on the heating rate used.

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TABLE 1

INVESTMENT MATERIALS USED IN THIS STUDY

Investment	Manufacturer	Batch No.
Baker Cristobalite Investment for Inlays	Baker and Co. Inc.	110173
Baker Hygroscopic Investment	Baker and Co. Inc.	705052
R and R Hygroscopic Investment	The Ransom and Randolph Co.	None
Whip-Mix Cristobalite Model Investment	Whip-Mix Corp.	0417101

TABLE 2

COMPOSITION OF INVESTMENT MATERIALS*
(Percent by volume)

Investment	$CaSO_4 1/2H_2O$	Cristobalite	Quartz	Other
A	35	35	2-3	15
B	50	30	5	15
C	35	5	50	**Iron oxide 10
D	35	40	--	25

* Composition obtained by petrographic anaylsis from the Constitution and Microstructure Section, National Bureau of Standards.

** Iron oxide, a part of 10%

TABLE 3

EFFECTS OF WATER-POWDER RATIO ON THE SETTING AND THERMAL EXPANSION OF INVESTMENT A

Water-Powder Ratio	Setting Expansion	Thermal Expansion	Total Expansion
	%	%	%
30/100	0.35	1.38	1.73
	.37	1.35	1.72
32/100	.35	1.25	1.60
	.35	1.18	1.53
34/100	.36	1.15	1.51
	.42	1.18	1.60
36/100	.35	1.12	1.47
	.37	1.23	1.60
38/100	.34	.98	1.32
	.36	1.04	1.40
40/100	.34	.98	1.32
	.34	.95	1.29

TABLE 4

EFFECT OF WATER-POWDER RATIO ON THE SETTING EXPANSION AND HYGROSCOPIC EXPANSION OF INVESTMENTS C AND D

Investment	Water-Powder Ratio	Setting Expansion (in air)	Hygroscopic Expansion (in water)
		%	%
C	32/100	0.32	0.65
		.36	.60
		.32	
	34/100	.29	.57
		.29	.56
D	34/100	.31	.62
		.37	.57
		.43	.61
		.44	.60
	38/100	.28	.51
		.26	.48
		.31	.49
		.38	.60

TABLE 5

COMBINED HYGROSCOPIC AND THERMAL
EXPANSION OF INVESTMENTS A AND B*

Investment	Expansions	Water-Powder Ratio					
		30/100	(30+2)/100	(30+4)/100	(30+6)/100	(30+8)/100	(30+10)/100
		%	%	%	%	%	%
A	Setting (Hygroscopic)	0.40	0.45	0.52	0.57	0.78	0.78
	Thermal (at 700°C)	1.28	1.25	1.20	1.19	1.14	1.02
	Total	1.68	1.70	1.72	1.76	1.92	1.80
B	Setting (Hygroscopic)	0.36	0.47	0.52	0.56	0.57	0.64
	Thermal (at 700°C)	1.11	.99	.94	.90	.90	.89
	Total	1.47	1.46	1.46	1.46	1.47	1.53

* The hygroscopic and thermal expansion data are the average of two or more test runs.

TABLE 6

EFFECTS OF ADDITION OF WATER DURING SETTING PERIOD, ON THE SETTING, THERMAL AND TOTAL EXPANSION OF INVESTMENT A

Expansions	Water-Powder Ratio			
	(25+5)/100	(27+3)/100	(29+1)/100	30/100
	%	%	%	%
Setting (Hygroscopic)	0.82	0.58	0.49	0.40
Thermal (at 700°C)	1.15	1.17	1.22	1.28
Total	1.97	1.75	1.71	1.68

TABLE 7

EFFECT OF THREE DIFFERENT HEATING RATES ON THE EXPANSION AT 700°C, OF INVESTMENT MATERIALS

Investment	Water-Powder Ratio	Heating Rates 3.75°C/Min	5.62°C/Min	11.25°C/Min
		%	%	%
A	25/100	1.40	1.37	1.34
		1.43	1.38	1.35
	30/100	1.27	1.25	1.30
		1.26	1.23	1.27
	38/100	1.04	1.16	1.04
		1.05	1.09	1.09
B	30/100	1.18	1.16	1.21
		1.14	1.13	1.12
		1.10	1.13	1.22
		1.07	1.11	
			1.07	
C	30/100	0.75	0.76	
		.65	.77	
		.60		
	34/100	.61	.56	
		.60		
		.63		
	38/100	.37		
		.38		
		.34		
D	34/100	1.14	1.17	
		1.06	1.13	
		1.15	1.12	
		1.10	1.15	

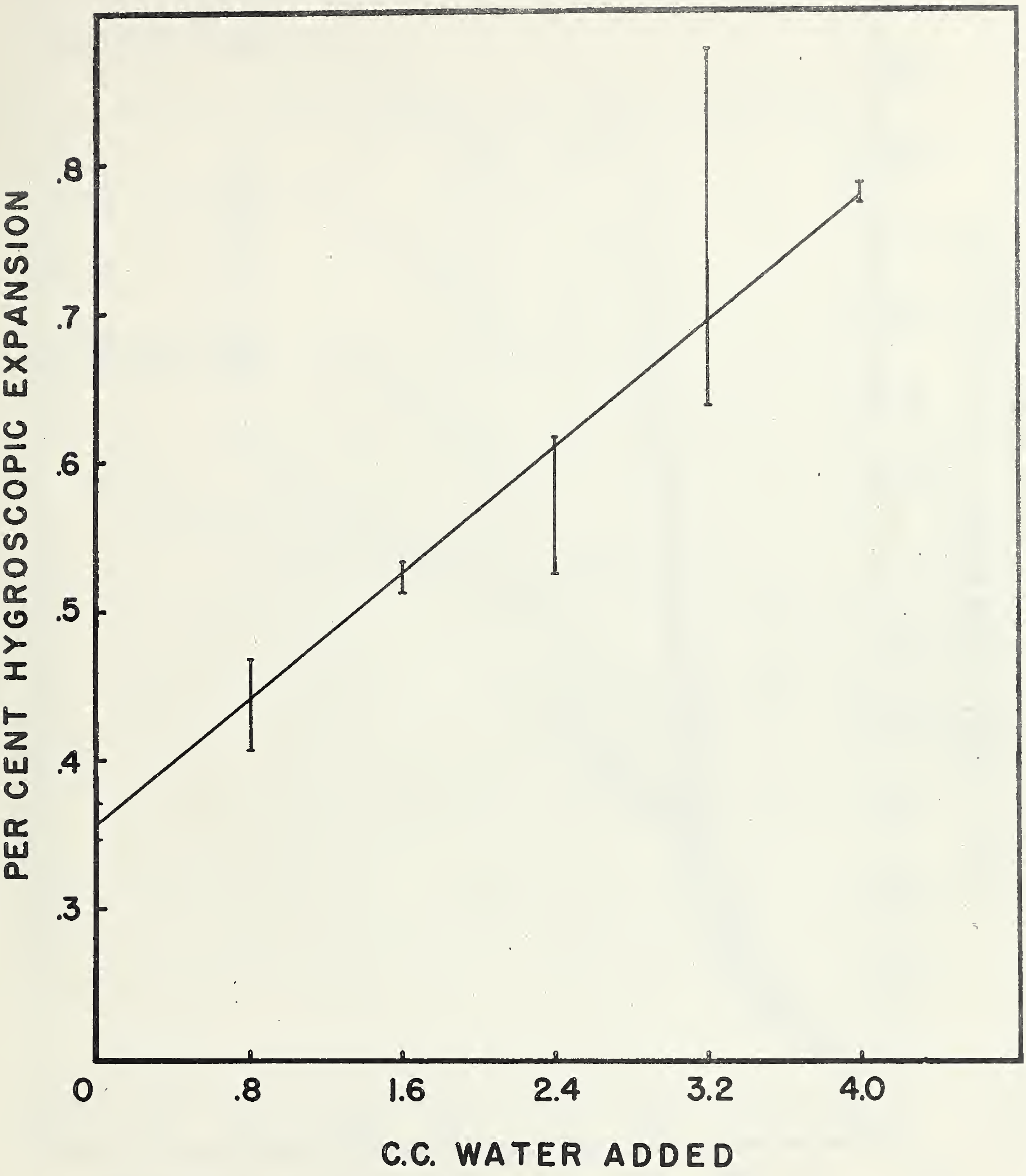

Figure 1: Hygroscopic expansion of investment A following the addition of increasing amounts of water.

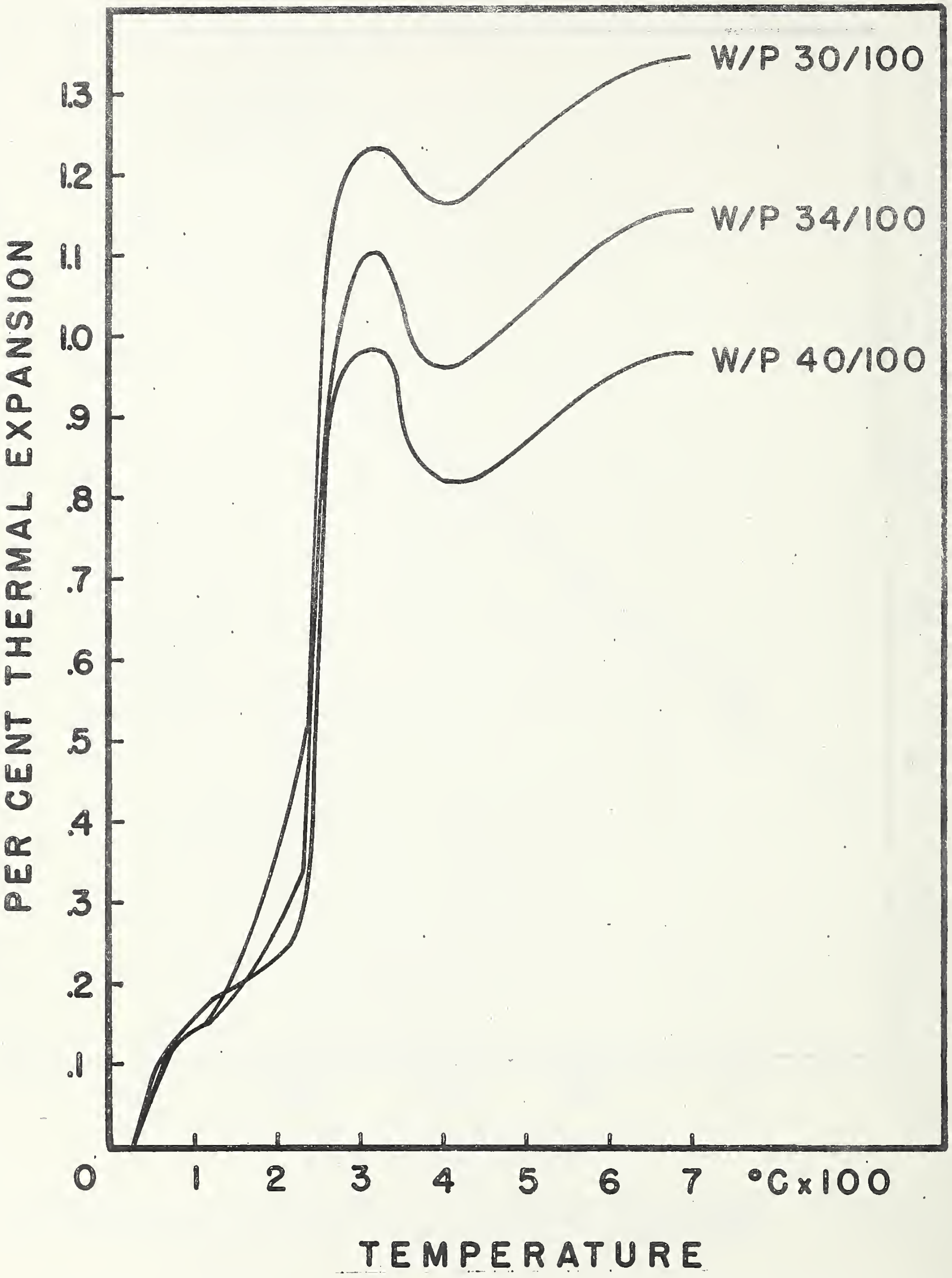

Figure 2: Effect of three different water powder ratios on the thermal expansion of investment A, a material containing a cristobalite refractory.

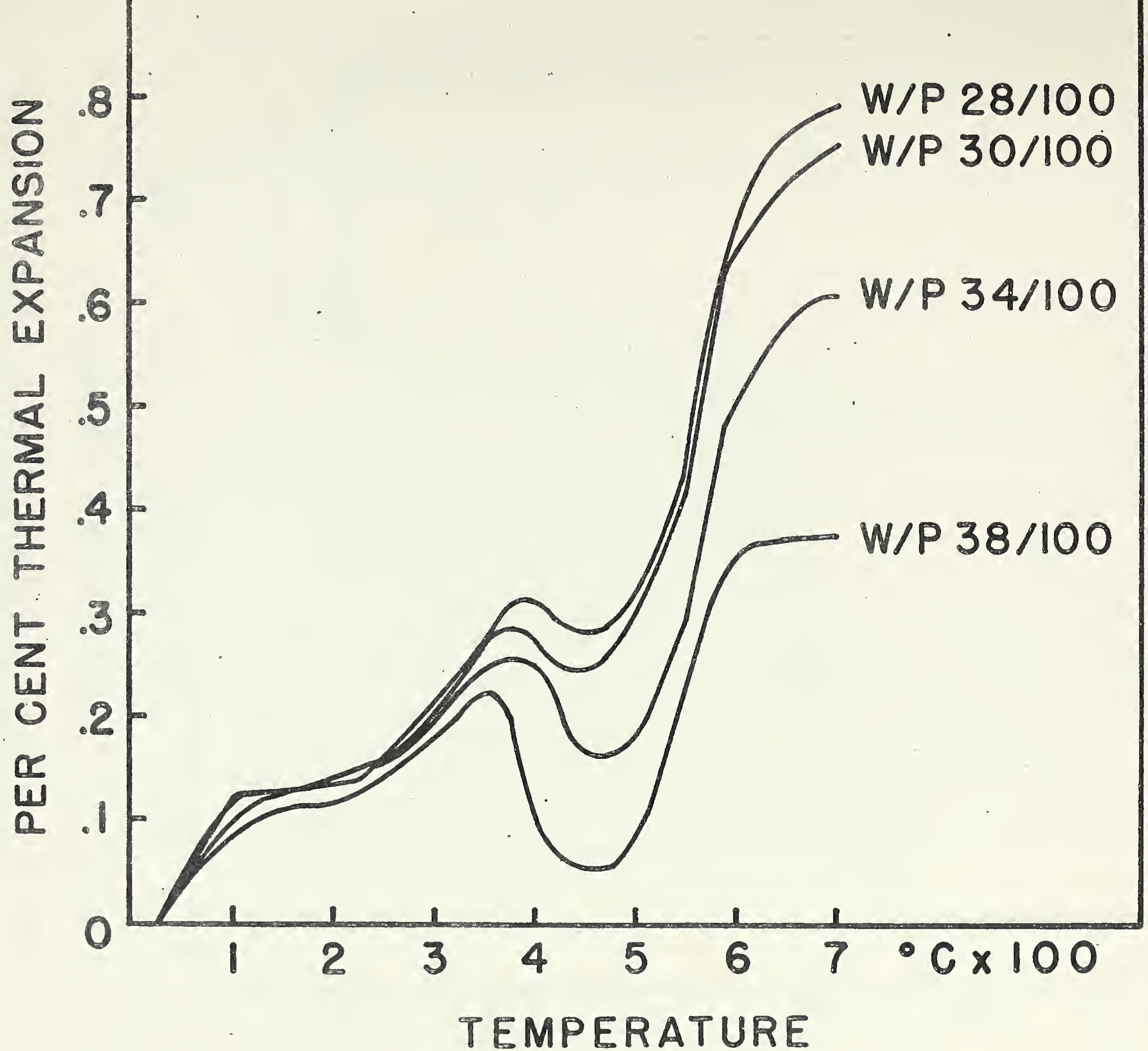

Figure 3: Effect of four different water-powder ratios on the thermal expansion of investment C, a material containing a quartz refractory.

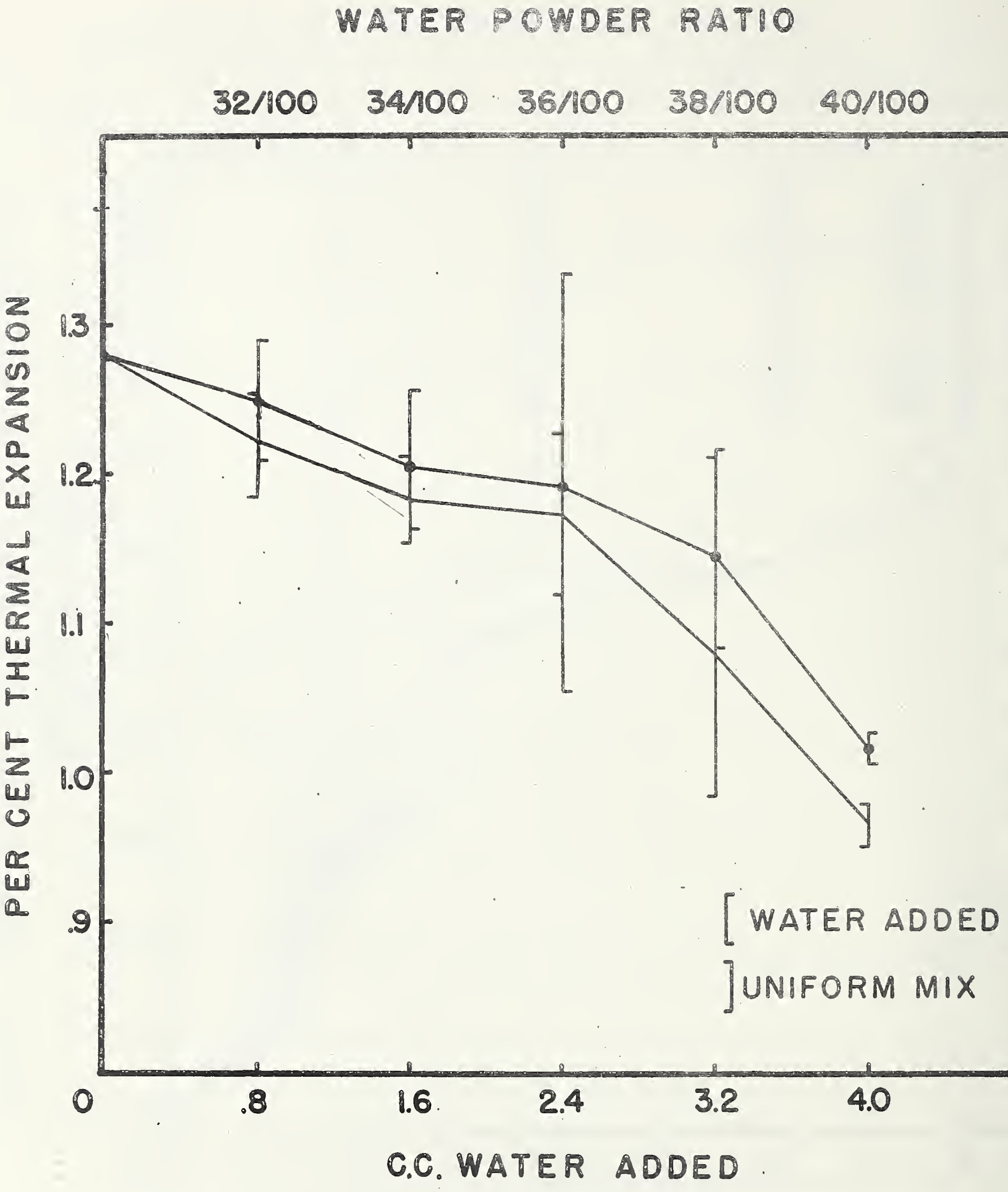

Figure 4: Comparison of the average thermal expansion at 700°C of investment A using definite water-powder ratios and hygroscopic expansion by water addition.

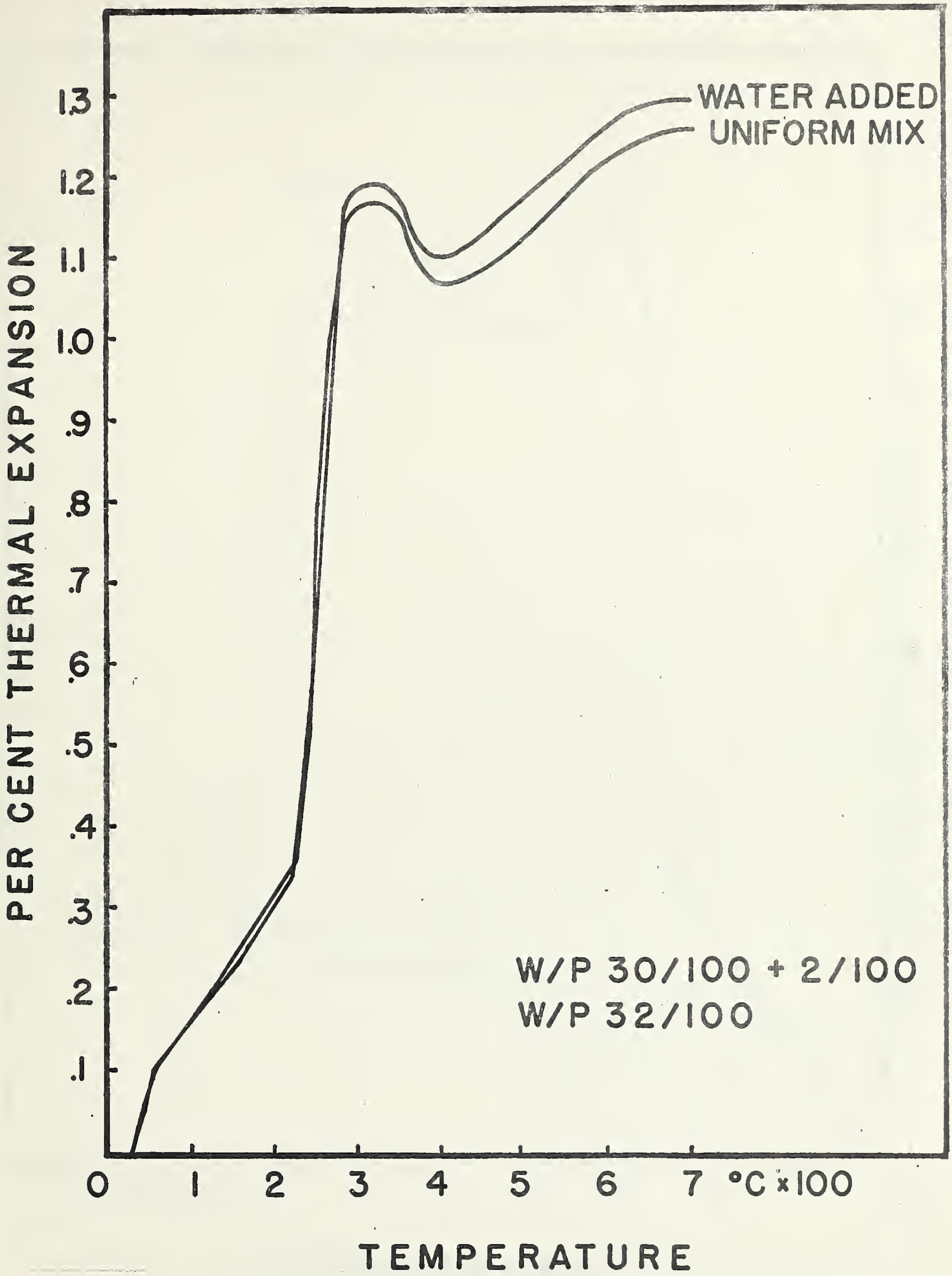

Figure 5: Comparison of the thermal expansion curves of a specimen of investment A with a water-powder ratio of 32/100 and of a specimen with initial water-powder ratio of 30/100 to which sufficient water to bring the ratio up to 32/100 was added to produce hygroscopic expansion.

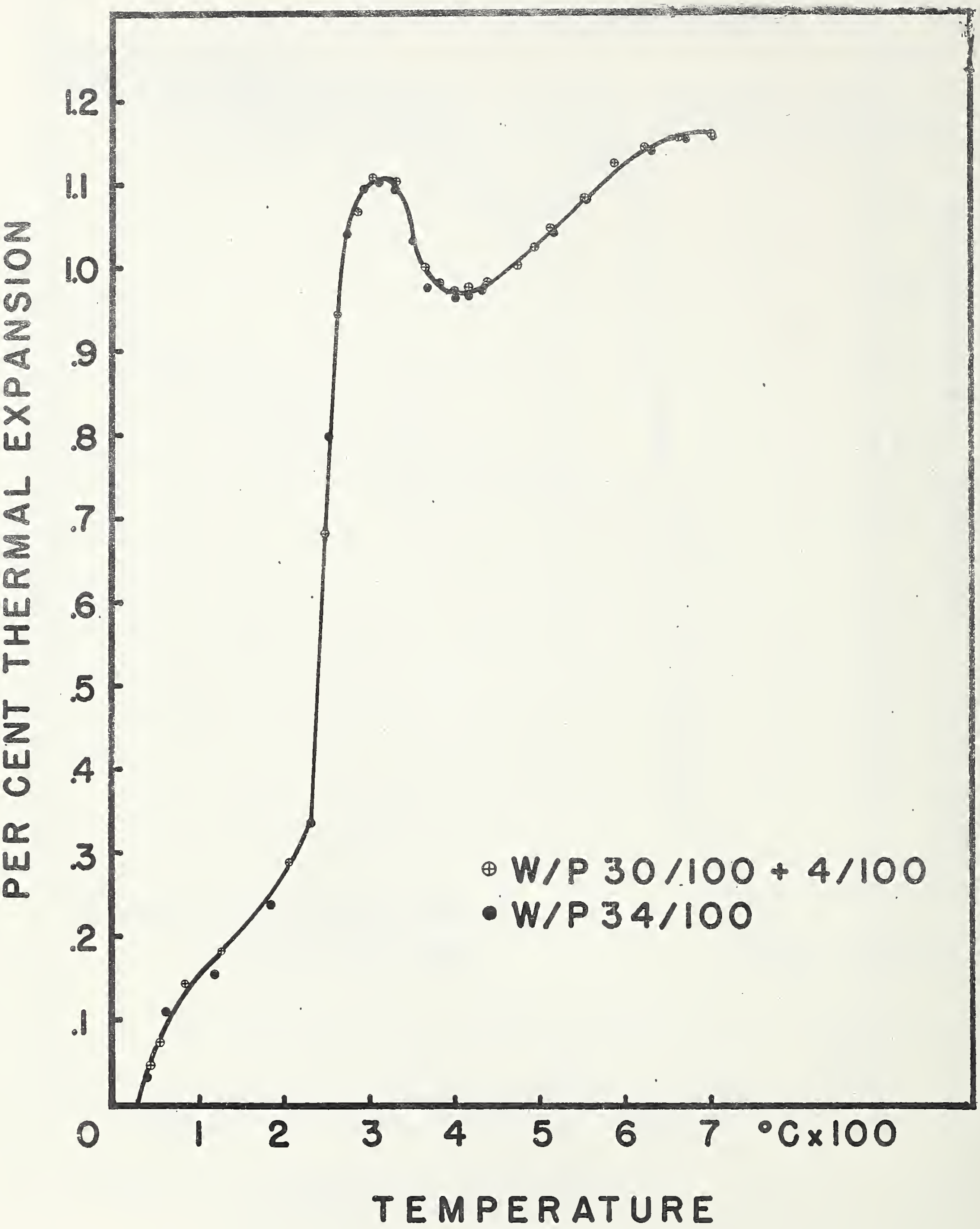

Figure 6: Comparison of the thermal expansion curves of a specimen of investment A with a water-powder ratio of 34/100 and of a specimen with initial water powder ratio of 30/100 to wh sufficient water to bring the ratio up to 34/100 was added produce hygroscopic expansion.

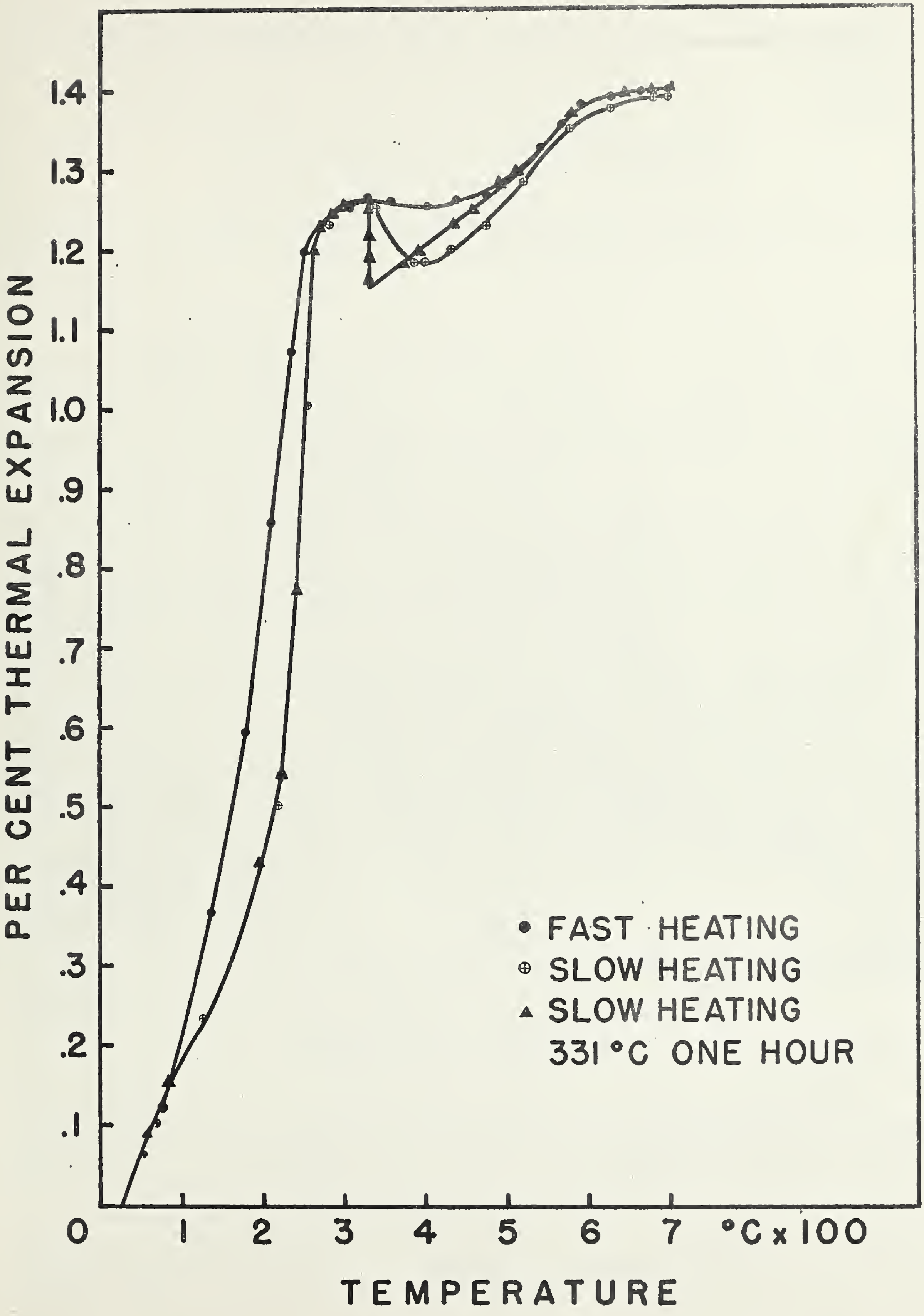

Figure 7: Effect of rates of heating on the thermal expansion of investment A.

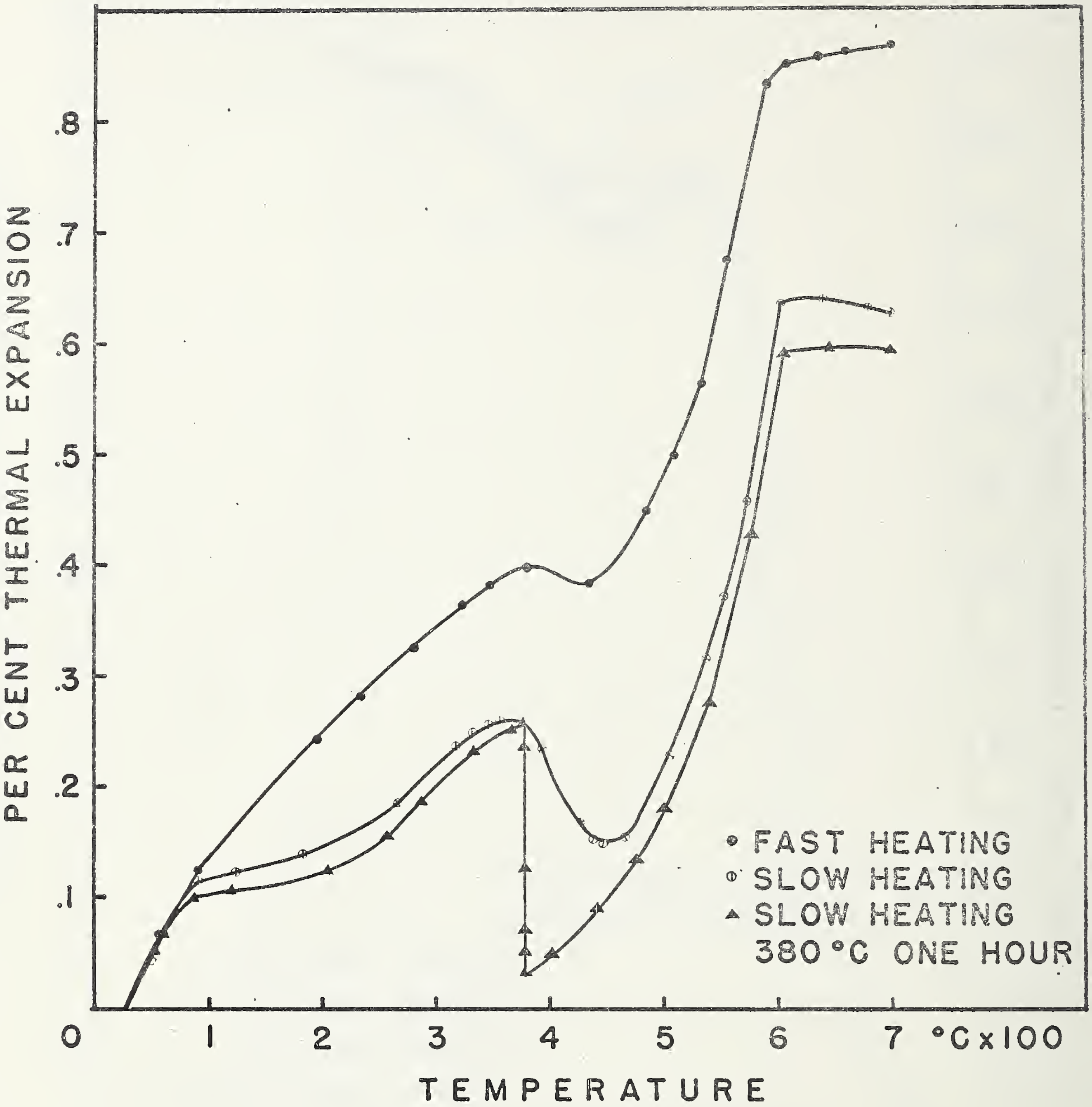

Figure 8: Effect of rates of heating on the thermal expansion of investment C.

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