Contrails

FOREWORD

This report was prepared by Armour Research Foundation, Chicago, Illinois under USAF Contract No. AF 33(616)-7687. This contract was initiated under Project No. 7351, "Metallic Materials", Task No. 735105, "High Strength Metallic Materials". The work was administered under the direction of the Directorate of Materials and Processes, Aeronautical Systems Division, with Mr. Paul L. Hendricks acting as project engineer.

This report covers work conducted from February 1961 to March 1962.

Armour Research Foundation personnel who made major contributions to the program were: F. A. Crossley, project leader; B. J. Stang, project technician; T. Niemczyk, project technician;and J. R. Dvorak, metallographer. The data reported herein are recorded in ARF Logbooks Nos. C-11165 and C-12212, assigned to Project No. ARF 2213. This report is identified internally as ARF 2213-14.

Approved for Public Release

Contrails

Approved for Public Release

Contrails

ABSTRACT

The objective of this program is the inhibition of beta grain growth in titanium alloys. The principal means investigated for accomplishing this objective was the addition of minor amounts of solutes. Grain growth inhibition was expected to result from the preferential adsorption of the additions on the grain boundary and the consequent lowering of the grain boundary energy. Solutes were selected on two bases: (1) experimental indication of effectiveness as reported in the literature, and (2) anticipated high degree of polarizability. Solutes of high polarizability were expected to be relatively more effective in lowering the grain boundary energy because of the ease by which their outer electronic field can be distorted. The study involved three base compositions: unalloyed titanium, B-120VCA (Ti-13V-11Cr-3Al), and Ti-8Al-1Mo-1V alloy.

Additions selected for high polarizability were barium and strontium. Other additions evaluated were boron, sulfur, and silver plus hafnium. The sulfur and boron additions produced particulate phases in the alloys. Only additions of sulfur or boron inhibited beta grain growth in titanium and Ti-8Al-1Mo-1V alloy. However, in B-120VCA barium was only slightly less effective in inhibiting grain growth than sulfur or boron.

Room temperature tensile properties gave no indications of detriment to mechanical properties due to any of the additions. Strontium appeared to be beneficial to ductility through a scavenging action.

This technical documentary report has been reviewed and is approved.

I Perlmutter

I. PERLMUTTER
Chief, Physical Metallurgy Branch
Metals and Ceramics Laboratory
Directorate of Materials and Processes

Approved for Public Release

Contrails

TABLE OF CONTENTS

Approved for Public Release

Contrails

LIST OF ILLUSTRATIONS

Approved for Public Release

Contrails

LIST OF ILLUSTRATIONS (Cont.)

Approved for Public Release

Contrails

LIST OF TABLES

Approved for Public Release

Contrails

Approved for Public Release

Contrails

THE EFFECT OF ADDITION ELEMENTS ON THE RATE OF BETA GRAIN GROWTH IN ALPHA-BETA AND BETA TITANIUM ALLOYS

I. INTRODUCTION

It has long been recognized that heating titanium-base alloys above the beta transus temperature results in loss of ductility that can only be recovered by subsequent working below the beta transus temperature. The cause of ductility loss is reportedly due primarily to large beta grain size (1). Large beta grain size permits large cracks to develop as a result of extensive slip on a few planes. This problem was solved by not heating alloys into the beta phase field. Newer developments, however, have returned this problem. Consider the following examples:

(1) The Ti-7Al-4Mo alloy is in demand in the extruded form. Although it can be extruded in the alpha plus beta field, this is difficult, whereas extrusion in the beta field is relatively easy. Also, it has been shown that the Widmanstätten structure obtained by heating into the beta phase field has creep-rupture strength and creep resistance substantially superior to the equiaxed alpha-beta microstructure obtained by working and solution treating below the beta transus (2).

(2) The recently developed "beta" age-hardenable alloy B-120VCA is annealed and solution heat-treated in the beta phase field. Grain growth is particularly a problem in this alloy.

Occasionally, titanium alloys that are customarily fabricated and heat treated below the beta transus are heated above it. This may be inadvertent or it may be intentional in order to facilitate fabrication. In the former case, ductility properties may be so seriously degenerated that the part is not usable. In the latter case ductility is decreased, and it would be desirable to keep such loss as low as possible.

The objective of this program is to inhibit grain growth in beta titanium through the application of two techniques: (1) alloy additions and (2) recrystallization and stabilization to a fine grain size at a relatively low temperature before heating to the ultimate temperature in the beta phase field desired. Minor amounts of six separate additives were made to three base compositions: unalloyed titanium (100 BHN hardness), B-120VCA (Ti-13V-11Cr-3Al), and Ti-8Al-1Mo-1V alloy.

Manuscript released by author April 1962 for publication as an ASD Technical Report.

Approved for Public Release

Contrails

II. LITERATURE REVIEW

A. Solute Additions for Grain Growth Inhibition

Titanium alloys (3) and many other metals (4) follow the grain growth relationship expressed in Eq. 1

$$D = Kt^n \tag{1}$$

where D is the average diameter, t is time, K is a constant and n is the growth rate exponent. Derivation of a growth rate expression may be based upon the following assumptions:

(1) The rate of grain boundary migration is inversely proportional to its radius of curvature.

(2) The sole driving force is grain boundary free energy.

(3) The grain boundary mobility and interfacial free energy are constant throughout the grain growth process.

(4) The geometry of grains changes only by a scale factor (there is no preferred direction of growth).

(5) Inclusions are absent or have no influence on grain boundary migration.

(6) The initial grain size is small compared to the final size.

This results in Eq. 1 with $n = 1/2$ (ref. 4). Bubbles in a soap froth obey this specific relationship. However, for metals and alloys n apparently varies over the approximate range from 0.1 to 0.7 (ref. 4). The value of n changes with both composition and temperature. The variation of n reflects changes in the grain boundary free energy due to solutes, temperature, and other effects. Since low values of n correspond to low grain growth rate, the latter property can be suppressed by proper selection of solutes.

There are ample representatives in the literature of solutes and impurities which segregate preferentially to grain boundaries. This follows from the fact that the grain boundaries are regions of greater disorder than the grains; as a consequence, the increase in energy due to the placement of a foreign atom in the metal lattice is lower at the grain boundary than in the grains proper. The arguments of Weyl (5) would lead to the expectation that solute additions of much higher polarizability or polarizing power than the solvent would lower the free energy of the grain boundary by lowering the potential field at the interface. Polarizability is the susceptibility of an ion to distortion of its electron field, while polarizing power is the ability of one ion to bring about distortion in the electron field of a neighboring ion. Lowering of grain boundary energy results from distortion of the electron field of either the solute ions concentrated therein or the solvent ions.

Approved for Public Release

Contrails

Quantitative measurements of polarizability of metals are not available. Polarizability is a derived quantity and is usually determined from dielectric measurements. However, the electrical means for the determination of dielectric constants are not applicable to conductors. Polarizability of metal ions having noble gas electronic configurations can be calculated roughly from the ion radius. However, this is based upon the assumption that the metal ion is in a state approximating that of a noble gas. The difference between this and the metallic state is that in the latter the atoms lose their identity by merging electrons in a joint electronic system. Because of this difference the electronic structure and, consequently, the polarizability of the ion bear no relationship to the metallic state. In principle, polarizability can be calculated from the optical properties index of refraction and index of absorption for nonmagnetic metals. However, the polarizability thus obtained varies with the angle of incidence and the wavelength of the light. Also, it is found experimentally that there is dependence on the surface condition of the metal and the particular technique used as well--e.g., measurement on massive metal as opposed to transparent films.

This impasse may be, to some extent, circumvented by considering two properties: atomic diameter and position in the Periodic Table. For elements and ions having the ideal gas structure polarizability is proportional to the cube of the atomic or ionic radius. Also, for elements of a given period the ionization potential increases with increasing atomic number. While polarizability is a measure of the ease with which the outer electrons of an atom or ion can be distorted, ionization potential is a measure of the binding strength of the most loosely bound electron. It is apparent that these two properties should bear some sort of a direct relationship. The more loosely the electron is bound, the more easily it can be distorted. These factors, together with the necessity of being able to alloy the element with titanium (at least, to a limited extent), led to the high polarizability candidates as given in Table I. Based upon the data given in Table I it is projected that the elements in order of decreasing polarizability are cesium, rubidium, barium, and strontium.

Berger et al. (6) found that sulfur suppresses grain growth in titanium. An addition of 0.02% reportedly decreased the tensile elongation of iodide titanium from 16% to 13%, while an addition of 0.2% decreased the tensile elongation of commercial purity base Ti-6Al-4V alloy from 16% to 13% and the reduction in area from 36% to 30%. (One questioning note is sounded by the fact that the elongation reported for the pure iodide titanium--in sheet form--is less than that expected from commercial (99%) purity material.)

Reinbach (7) investigated the recrystallization of unalloyed titanium and the effectiveness of the elements Ag, Al, Cb, Co, Cr, Cu, Fe, Ge, and Hf in retarding recrystallization. The writer's experience with silver additions to titanium alloys suggests that small quantities would not be detrimental to mechanical properties.

Winter and Weinig (8) found that grain boundary shear during elevated temperature creep was significantly decreased by solutes adsorbed on the grain boundary in dilute titanium alloys. Concomitantly, the activation energy for the grain boundary stress relaxation phenomenon was increased

Approved for Public Release

Contrails

as a function of the solvent-solute misfit for binary alloys. They suggest that a much greater restraint to grain boundary shear could be obtained from the use of a spectrum of solutes of various atomic sizes. Additions up to 0. 12 at. % of Cb, Au, Al, V, and Zr were made. These elements had increasing effect on the activation energy for grain boundary relaxation in the order given. It seems reasonable that there would be correlative effect on grain growth.

Very small insoluble particles can be very effective in inhibiting grain growth; however, such particles are much more likely to be detrimental to the mechanical properties of highly alloyed titanium than soluble additions. In this regard boron was reported to be the most promising addition investigated in programs conducted several years ago having the same objective as the current program (9, 10).

B. Fine Grain Size Stabilization as a Means of Growth Control

Lucke and Detert (11) state that the driving force for grain growth is smaller than that for recrystallization by a factor of about one thousand. This fact, together with the author's observations on grain growth of recrystallized beta brass, suggests a different means for controlling grain growth. It was observed that beta brass, extruded at 300°C (572°F), exhibited much larger grain size when annealed at temperatures of 800°C (1472°F) and 825°C (1517°F) for 24 hours than when first annealed at 400°C (752°F) for 1/2 hour before the high-temperature treatment. The 400°C anneal resulted in a very fine, uniform grain structure. The procedure suggested is to recrystallize at a low temperature to produce a stable, fine-grained structure before heating to the desired temperature above the beta transus. For alpha-beta titanium alloys the temperature for grain size stabilization is preferably just below the beta transus. In this way, residual small islands of alpha will inhibit beta grain growth (12). Obviously, if this technique is found to be effective, it could easily be combined with the solute control technique.

III. SELECTION OF SOLUTES

The solute candidates for grain growth inhibition are presented in Table I. They are divided into two groups. The first group are those that have been shown by experiment to have promise; the second group are elements anticipated to be highly polarizable because of their large atomic size and their low ionization potential. From the standpoint of polarizability rubidium and cesium were considered to be the most promising, in that order. However, attempts to prepare master alloys of cesium were unsuccessful. (Since cesium and rubidium melt at temperatures less than 105°F and boil at temperatures of less than 1300°F, prealloying would be an absolute necessity in order to add them to titanium alloys which, of necessity, are prepared in arc furnaces.)

Approved for Public Release

Contrails

The final selection of additions for grain growth inhibition are given below:

Number	Element(s)	wt %	at. %
1.	sulfur	0.05	0.07
2.	silver, hafnium	0.45 0.74	0.2 0.2
3.	barium	0.57	0.2
4.	strontium	0.36	0.2
5.	boron	0.11	0.5
6.	sulfur, hafnium, barium, strontium, boron	0.027 0.15 0.11 0.073 0.0090	0.04 0.04 0.04 0.04 0.04

The additions 1, 2, and 5 were selected on the basis of reports in the literature of effectiveness in suppressing grain growth in titanium. Additions 3 and 4 were based upon the high polarizability criterion tempered by practical necessity of getting the addition into the titanium solid solution. The sixth addition was made up of five elements representing a spectrum of atomic sizes based upon the results of Winter and Weinig (8).

IV. EXPERIMENTAL METHODS

The sources, form, and purity of the materials used in this investigation are summarized in Table II. The major alloying additions aluminum and chromium were added in the elemental form, while all other additions were made in the form of master alloys. A Mo-22.5Al master alloy was prepared by melting in an nonconsumable electrode arc furnace. The V-15Al master alloy was purchased as such, see Table II. The minor additions for grain growth control were prepared as binary titanium-base alloys containing 25 at.% of the solute except for barium and strontium, which were prepared as ternary alloys containing 47.5 at.% solute, 5 at.% titanium, and 47.5 at.% aluminum.

Considerable difficulty was experienced in making master alloys of the low polarizability additions. The initial choices for these were cesium and rubidium. These metals have melting points of 83° and 102°F, respectively, are extremely reactive to air, and are explosively reactive to moist air. Therefore, the only practical means of adding them to titanium, which

Approved for Public Release

Contrails

has to be arc melted, is to alloy them to increase the melting temperatures and the oxidation resistance. The first attempt to prepare a Ti-Cs master was carried out by mixing titanium powder and liquid cesium in a Vycor bulb under argon. The bulb was stoppered and heated to 200°C (390°F) for 48 hours. There was no evidence of alloying. For the second attempt aluminum and titanium powders were added to cesium in a Vycor bulb, and the stoppered bulb was heated to 200°C (390°F) for 24 hours, 400°C (750°F) for 54 hours, and then to 450°C (840°F) for 30 hours. Upon examination it was found that the cesium had reacted with the bulb. The final trial consisted of heating cesium, aluminum, and titanium powder together in a Vycor bulb at 200°C (390°F) for 24 hours followed by 450°C (840°F) for 4 1/2 hours. Subsequent examination of the contents of the bulb at room temperature showed the presence of a liquid phase, indicating no meaningful alloying from the standpoint of the needs of the program.

After these attempts barium and strontium were substituted for cesium and rubidium. Because of the low alloying level it was desired to make the additions in relatively small particles in order to have greater assurance of producing homogeneous alloys. Consequently, alloying was again a necessity because of the low oxidation resistance of these metals. The first attempt to produce a Ti-Ba master was to melt barium rod together with titanium sponge in a nonconsumable electrode arc melting furnace. This attempt was unsuccessful due to the high vapor pressure (i. e., low boiling point) of barium. The second attempt was made melting barium rod and titanium sponge together in a Vycor bulb at 750°C (1380°F) for 24 hours. The barium reacted with the bulb. Success was finally achieved by melting barium together with 47.5 at.% aluminum and 5 at.% titanium in a graphite crucible under argon, the heat being supplied by induction. The strontium master alloy was prepared in the same manner.

The Ti-7.0B* master alloy was prepared by melting in a nonconsumable arc-melting furnace. For this operation high-purity boron powder was first consolidated by compacting, sintering, and finally arc-melting.

The Ti-18.2S master alloy was made by heating -1/16 mesh titanium sponge together with roll sulfur in an evacuated Vycor bulb from room temperature to 450°C (842°F), holding for 24 hours and then heating to 750°C (1382°F) and holding for 1 hour. An initial Ti-S melt, prepared similarly but heated to a higher final temperature of 950°C (1742°F), was discarded because of contamination; the bulb fractured during the thermal treatment. The initial step in the preparation of the Ti-43Ag master alloy was carried out by similarly heating to 975°C for 4 hours. The product was then homogenized by melting in a nonconsumable electrode arc furnace. The Ti-55Hf alloy was prepared by melting in a nonconsumable electrode arc furnace.

The master alloys were particulated by crushing, except for the hafnium alloy which was machined into chips since it was too ductile for crushing.

---

* Alloy designations are in weight per cent.

Approved for Public Release

Contrails

Five-pound ingots of the alloys were prepared by double arc melting. Ingots resulting from initial melting in a nonconsumable arc furnace were quartered longitudinally and the segments welded end-to-end to provide an electrode for the final consumable electrode arc melting.

The Ti-0.57Ba alloy did not melt properly in the second (consumable electrode) melting operation. A hollow metal shell was produced. The shell was cut into pieces preparatory to remelting in a nonconsumable-electrode arc furnace. However, when it was determined that the pieces would have to be further sectioned to dimensions less than 1/2 in. in order to feed them into the furnace, it was decided to abandon this melt. The further cutting would have required too much time. The B-120VCA alloy with barium added behaved in the same way in the consumable electrode arc-melting operation. However, the problem of remelting was attacked in a different way. The shell was quartered longitudinally, welded into an electrode, and remelted in the consumable electrode arc furnace.

The ingots were dressed by grinding and then forged to plate 1 3/4 in. thick. Forging temperatures for the unalloyed titanium group, the B-120VCA group, and the Ti-8Al-1Mo-1V group were 1600°, 2200°, and 2000°F, respectively. The forged plates were sandblasted and surface ground preparatory to rolling. The rolling schedule of the alloys is given in Table III. Rolling was done on a two-high Bliss mill with 10 in. diameter by 14 in. length rolls. Initial attempts to roll the B-120VCA group at 1500°F were unsuccessful, and it was necessary to raise the temperature to 1800°F. After rolling to 0.120 in. the materials were sandblasted and pickled to remove about 5 mils from the total thickness. Specimens approximately 3/8 x 5/8 in. were cut for the grain growth anneals. These specimens were stress relieved by annealing at 1300°F--for 1 hour in the case of unalloyed titanium group and 2 hours for the Ti-8Al-1Mo-1V group.

Materials with "stabilized" grain structures were prepared by taking rolled materials at the 1/4 in. thick stage, sand blasting, pickling off 5 mils total from the thickness and cold rolling to 0.120 in. in passes of 5 mils. Cold reduction was 52%. Pickling solution used throughout this program was 3% HF, 30% HNO_3, balance water at 150°F. Alloys prepared with "stabilized" structures were the base unalloyed titanium, B-120VCA, and these two materials containing the 0.05% sulfur addition. Stabilization anneals were 1100°F-24 hr and 1250°F-24 hr for the unalloyed titanium and the B-120VCA alloys, respectively.

Specimens were sealed in evacuated Vycor bulbs--together with a few particles of sponge titanium for gettering--for heat treatments. These treatments varied in temperature over the range from 1700° to 2100°F and in time from 1/4 to 24 hours. All specimens were water quenched from grain growth anneals.

Tensile test specimens were prepared from materials further reduced to 0.065 in. The unalloyed titanium, B-120VCA, and Ti-8Al-1Mo-1V groups were rolled in passes of 0.025 in. at temperatures of 1300°, 1700°, and 1800°F, respectively. After sandblasting and pickling, tensile specimens of the geometry

Approved for Public Release

Contrails

shown in Fig. 1 were prepared. Tensile specimens of the B-120VCA and Ti-8Al-1Mo-1V groups were sealed in evacuated Vycor bulbs for heat treatments.

Tensile tests were performed on a 10,000-lb Instron unit at a cross-head speed of 0.1 in./min. Cal-Tester sheet grips were employed in these tests. Stress-versus-extension curves were plotted autographically.

Grain size determination was by the intercept (or Heyn) method--ASTM Designation E91-51T. For grain sizes less than about 0.01 in. diameter at least 40 grains were included in each determination. It was usually not possible to count this many grains when the grain size was larger than this.

Specimens were prepared for metallographic examination by electrolytic polishing following the procedure described by Adenstedt et al. (13) using an electrolyte of the following composition (14): 590 ml methyl alcohol, 350 ml butyl cellosolve (2 butoxyethanol) and 60 ml perchloric acid (7%).

Spot check chemical analysis are reported in Table IV. The results are in fair agreement with the nominal compositions except with respect to chromium. The low chromium results are attributed to the fact that it was added as a rather fine (50 mesh) powder. Analytical procedures for the minor additions in the presence of titanium were not readily available. Since development of such procedures was clearly beyond the scope of this program, no attempt was made to analyze for these.

Vacuum fusion analyses for the gases hydrogen, nitrogen, and oxygen of several tensile test specimens are reported in Table V. The oxygen content of the B-120VCA base alloy appears to be particularly high, while the nitrogen contents of this alloy and of the Ti-8Al-1Mo-1V + 0.36Sr alloy appear to be especially low.

V. RESULTS AND DISCUSSION

A. Microstructures

Microstructural conditions at the start of grain growth anneals for the three groups: unalloyed titanium, B-120VCA, and Ti-8Al-1Mo-1V are presented in Figs. 2, 3, and 4, respectively. The alloys containing minor additions of sulfur or boron contain particles due to these additions. Also, it may be noted that the base titanium contained second phase particles. There was no evidence that barium or strontium introduced a new phase in the alloys.

Approved for Public Release

Contrails

In the case of the unalloyed titanium group, some of the alloy microstructures were photographed under polarized light when this resulted in better definition of the grain structure. Considering this group, finest initial grain size occurred in the sulfur-containing alloy and largest grain size occurred in the strontium-containing alloy. The average grain diameter of the latter was three times that of the former.

Comparison of the initial microstructures of the B-120VCA group shows that the alloys containing a particulate phase due to sulfur or boron and the alloy containing the 0.57Ba addition have the finer grain sizes. The 0.57Ba alloy exhibited the finest grain size, the alloys containing boron additions were of slightly greater grain size, and the 0.45Ag-0.74Hf alloy had the coarsest grain size, the average grain diameter being 2.4 times that of the finest grained alloy.

Initial microstructures of the Ti-8Al-1Mo-1V group show finest grain size for the 0.11B addition and coarsest grain size for the 0.57Ba addition.

B. Effect of Minor Additions on Grain Growth

While it is interesting to compare the effects of the additions in the initial microstructures, their real significance lies in the grain growth data. These data are presented in Tables VI, VII, and VIII, for the unalloyed titanium, B-120VCA, and Ti-8Al-1Mo-1V groups, respectively. There are a number of omissions in the tables. These omissions are the result of loss of identification numbers which were scribed on the specimens. Loss was due to surface evaporation or to welding of specimen surfaces one to another during the high-temperature anneals especially those of long duration. Some irregularity in grain growth resulted in alloys containing particles due to their non-uniform distribution. One of the requirements for accuracy in correlating grain growth data is that the smallest dimension of the specimen should not be smaller than about ten times the average grain diameter in order to avoid specimen size effects (15). This means a grain size limit of 10×10^{-3} in. for the 0.1 in. thick specimens of this investigation. It may be noted that only a few of the data fall below this limiting grain size. Also, growth rate expressions of the type exemplified by Eq. 1 are based upon the assumption that neither a second phase nor inclusions which influence grain boundary migration are present.

These conditions lead to the expectation that the grain growth data are not susceptible to quantitative analysis. The validity of this expectation is illustrated by Fig. 5 in which a log-log plot of average grain size versus time of annealing for unalloyed titanium is presented. Note the wide scatter about the lines (which were determined by the method of averages) and the nonsystematic variation in the slope of the curves with increasing temperature.

Qualitative comparisons of the relative effectiveness of the various additions in inhibiting grain growth in the beta phase field are shown in Figs. 6 through 17.

Approved for Public Release

Contrails

Figures 6 through 9 present the mean grain size of the alloys of the unalloyed titanium group plotted against annealing temperature for annealing times of 1/4, 1/2, 1, and 2 hours. Alloys which contain a particulate phase due to a minor addition are represented by closed symbols, while alloys in which the addition is soluble are represented by open symbols. It is at once apparent that significant grain growth inhibition occurs only in the alloys containing a particulate phase due to the addition. There are no data for the 0.57Ba addition since this alloy was lost in the second melting operation. It is interesting to note that the additions 0.05S and 0.11B were more effective than the multiple addition 0.03S-0.12Ba-0.15Hf-0.07Sr-0.01B, indicating that within the compositional range of this investigation the quantity of these two elements is important. The additions 0.45Ag-0.74Hf, and 0.36Sr appear to have no beneficial effect in inhibiting grain growth of unalloyed titanium above the beta transus temperature in the temperature range from 1700° to 1850°F. For the longer times of 1 and 2 hours at the highest temperature, the 0.45Ag-0.74Hf addition apparently promotes grain growth.

Figures 10 through 13 compare grain growth data for the B-120VCA group. For short annealing times of 1/4 and 1/2 hour the additions in order of decreasing effectiveness are: (1) 0.11B; (2) 0.05S, 0.57Ba, and the multiple addition; (3) 0.36Sr; and (4) 0.45Ag-0.74Hf. It is particularly interesting that the 0.57Ba addition appears to be competitive in B-120VCA alloy with the additions which produce particulate phases. For the longer times of 1 and 2 hours the 0.45Ag-0.74Hf addition apparently promotes grain growth at the higher annealing temperatures as in the case of unalloyed titanium.

Figures 14 through 17, which show grain growth data for the Ti-8Al-1Mo-1V group, clearly indicate a superiority for additions producing a particulate phase. These results are in agreement with those for unalloyed titanium. This behavior may be rationalized in terms of the initial microstructures. In the case of the alpha and alpha-beta structures of unalloyed titanium and Ti-8Al-1Mo-1V, the beta grains begin growing before the beta transus temperature is passed because of the phase-equilibrium requirements. (The unalloyed titanium of this investigation contains a small amount of beta due to the impurities present.) That is, beta grain growth is not controlled by the alpha grain boundaries, where the soluble type additions for grain growth inhibition may be expected to be concentrated. The beta grain boundaries move too rapidly for the soluble additions to segregate to them, and therefore, their motion is not influenced by them. Contrarily, the B-120VCA alloy is initially in a condition resulting from annealing in the beta field; consequently, solutes were given time to segregate preferentially to the grain boundaries.

Grain growth data for alloys "stabilized" by cold working and holding for a relatively long time at the recrystallization temperature are given in Table IX. Grain growth versus temperature for annealing times of 1/4 and 1/2 hour are plotted in Figs. 18 through 21. In the case of unalloyed titanium and this material with 0.05S added, the stabilization anneal of 1100°F-24 hr was below the beta transus temperature. Figure 18 indicates that for 1/4 hour annealing time stabilization benefited the base material

Approved for Public Release

Contrails

but is associated with larger grain size in the 0.05S-containing material. Comparison of the data in Table IX shows that for longer annealing times of 2 and 4 hours, there was no significant difference in grain size between the standard and stabilized conditions of the base material; while the standard condition of the 0.05S alloy exhibited much greater resistance to grain growth than the stabilized condition.

Grain growth data for the B-120VCA base alloy and for B-120VCA-0.05S in the standard and stabilized conditions are plotted in Figs. 20 and 21. The 1250°F-24 hr stabilizing treatment failed to produce primary recrystallization in the beta alloys. In the initial conditions of the alloys, the grains were elongated characteristic of cold-worked material. Note that after 1/4 hour at 1800°F the grain sizes are smaller than the initial grain sizes for the stabilized materials. The change to smaller and equiaxed grains means that recrystallization occurred. Hardness measurements of stabilized materials revealed no systematic variation in the values for specimens in the initial condition compared with specimens annealed at 1800°F for 1/4 hour. Hardness values were as shown below.

Material	Condition	Hardness (10 kg) DPH
B-120VCA	initial	275
	annealed 1800°F-1/4 hr	269
B-120VCA-0.05S	initial	284
	annealed 1800°F-1/4 hr	290

These hardness data indicate that stress relief was complete. Since recrystallization occurred during the grain growth anneal, the grain size of the stabilized specimens is smaller than would be expected for grain growth without simultaneous recrystallization. For annealing times of 1/2 hour the stabilized base material exhibited finer grain size for the most part, while in the 0.05S-containing material there was no substantial difference in grain size.

Comparison of data for 2- and 4-hour annealing times shows essentially the same grain size for the base alloy, except for annealing at 1900°F which resulted in significantly finer grain size for the stabilized material. Data for the 0.05S material was quite similar showing no substantial difference in grain size between the standard and the stabilized specimens except for annealing at 1900°F which resulted in finer grain size for the stabilized material.

It appears that the stabilization treatments imposed in this investigation were of little benefit to unalloyed titanium in inhibiting grain growth. The effect on B-120VCA is not clear. It would be necessary to evaluate a stabilization treatment which produces complete recrystallization in order to make a valid test. Under the present conditions the grain size was biased in the direction of being too small for early reaction times due to the formation of new grains by recrystallization.

Approved for Public Release

Contrails

C. Results of Microprobe Analysis of Alloys

Employing the best possible methods for obtaining good resolution, no differences in concentration were observed in either grain or grain boundaries of the major alloy constituents or the minor additions. All of the X-ray image photos showed a uniform distribution of the element in question. Samples representing thirteen of the twenty alloys studied were examined.

A sample of the base B-120VCA alloy was analyzed quantitatively. The results are not conclusive because of the inability of the spectrometer to resolve the X-ray lines of vanadium and chromium when they are both present in a titanium-base alloy. The results obtained (from Table IV), together with the wet analytical results, were as follows:

Element	Content, wt. %	
	Microprobe	Wet Analysis
Ti	75.6	79.5*
Al	2.1	3.26
V	17.1	11.7
Cr	5.3**	5.53
	10.0	

* By difference.

** These two values were determined by independent means; note that the smaller value is in good agreement with the wet analytical result.

Attempts to obtain quantitative analysis of the minor additions were unsuccessful.

D. Effect of Minor Additions on Tensile Properties

Tensile properties of the unalloyed titanium group are given in Table X. Tensile ductility was markedly uniform from alloy to alloy in this group. The lower strength properties of the alloys containing boron or strontium (that is, the 0.11B, the 0.36Sr alloy, and the multiple addition alloy) suggest that these elements have scavenging effects in titanium.

Specimens of the B-120VCA group were tensile tested in two conditions of heat treatment: (1) solution treated 1450°F-20 min-AC, and (2) solution treated and aged at 900°F-40 hr-AC. All specimens exhibited ductilities much inferior to that expected for the solution-treated condition: all specimens were brittle in the aged condition. It was found that the embrittlement was due to surface contamination. The degree of contamination is indicated by Fig. 22 which is a section from tensile test specimen B1-1. Note the surface crack which extends through the visible region of contamination. While it was known that B-120VCA picked up contaminants during processing more rapidly than the older alpha and alpha-beta alloys,

Approved for Public Release

Contrails

the degree of this greater propensity to absorb the interstitially soluble elements was not appreciated. As a consequence, insufficient surface material was removed in cleaning and pickling operations. Microhardness measurements on the B1-1 test specimen gave hardness values at the edge, at the center of the banded region denoting contamination, and at the center of the cross section of 523, 382, and 337 DPH, respectively.

The tensile properties of the Ti-8Al-1Mo-1V group are particularly interesting in that the base composition exhibited the poorest ductility for the heat-treated condition 1400°F-24 hr. Oxygen analyses for this alloy and for the 0.36Sr alloy were 0.199 and 0.155%, respectively. These are rather high contents when combined with 8% aluminum from the standpoint of ductility and, presumably, are the result of contamination during processing. It is worthy of note that the 0.36Sr alloy exhibited the highest ductility in all of the heat-treated conditions evaluated. It should be noted also that the 0.36Sr alloy analyzed 87% less nitrogen than the base alloy (see Table V). Note especially the 12.4% average elongation for the 0.36Sr alloy in the 1400°F-24 hr condition compared with 1.2% average elongation for the base alloy. These data too suggest a scavenging role for strontium in titanium.

Samples from several tensile test specimens were evaluated for surface contamination by microhardness measurements 1 and 5 mils from the surface, and at the center of the thickness. The results are given in Table XIII. These data indicate surface contamination only in those specimens which were solution treated in the beta phase field.

VI. SUMMARY AND CONCLUSIONS

Selected minor additions were made to titanium and to the titanium alloys B-120VCA and Ti-8Al-1Mo-1V for the purpose of inhibiting grain growth in the beta phase field. Selections were based upon one or the other of two criteria: (1) reports in the literature indicating or suggesting effectiveness, and (2) high polarizability. Polarizability is the property of an atom or ion which measures the ease by which its outer electronic structure can be distorted by an electric field. The regular array of atoms in a crystal lattice produces a uniform electric field. This field projects beyond the surface of the crystal giving rise to surface free energy. The adsorption of highly polarizable atoms on this surface lowers the energy by distortion of their outer electrons and thereby reduces the extent of the projection of the potential field. Therefore, in a polycrystalline array, solutes of high polarizability would be expected to lower the grain boundary energy and, as a consequence, reduce its rate of migration. Additions selected on the basis of high polarizability were barium and strontium. The other additions evaluated were: sulfur, silver plus hafnium, and boron. The additions were added in the amount of 0.2 at.%, except sulfur which was

Approved for Public Release

Contrails

added in the amount of 0.07 at.% and boron which was added in the amount of 0.5 at.%. A multiple addition made up of 0.04 at.% each of sulfur, hafnium, barium, strontium, and boron was also evaluated.

Boron and sulfur produced particulate phases in the alloys; while barium, strontium, silver, and hafnium were, apparently, in solid solution. Prior to the beta annealing heat treatments, the titanium and the Ti-8Al-1Mo-1V base alloys were stress-relief annealed below their beta transus temperatures, while the B-120VCA alloys were annealed above the beta transus temperature. Of the titanium and Ti-8Al-1Mo-1V alloys only those containing particulate phases due to sulfur, boron, or both exhibited marked resistance to beta grain growth. However, in the case of the B-120VCA alloys the 0.57Ba addition was approximately as effective in inhibiting grain growth as the additions sulfur and boron. Based upon ionization potential barium was considered to have a greater polarizability than strontium which did not appear to be effective in controlling beta grain growth.

The lack of effectiveness of barium in titanium and the Ti-8Al-1Mo-1V alloy may be explained in the following way. In these materials some beta is always present and, therefore, the beta grains start to grow before the beta transus is reached. This growth is very rapid since its driving force is the free energy of the phase transformation from alpha to beta. Under these conditions the solute has not time to segregate to the beta grain boundaries, and therefore, cannot be effective in inhibiting beta grain growth. Contrarily, a stabilized condition in which the soluble minor addition is segregated to the grain boundaries was achieved in the B-120VCA alloys prior to the grain growth anneals.

The multiple addition containing 0.2 at.% total of 5 additions was less effective than single additions of 0.07 at.% sulfur, or 0.5 at.% B. For beta grain growth inhibition in unalloyed titanium, sulfur was most effective at temperatures below 1800°F; boron was the most effective in the alloys B-120VCA and Ti-8Al-1Mo-1V and in unalloyed titanium at 1850°F. The lowest temperature of grain growth anneals for the B-120VCA and Ti-8Al-1Mo-1V alloys was 1800° and 1900°F, respectively.

Low-temperature recrystallization prior to application of grain growth anneals did not produce a stabilizing effect to retard grain growth. This possibly followed from the presence of stored energy of cold work retained in the recrystallized structures.

Room-temperature tensile properties indicated no detriment to mechanical properties by any of the additions. The ductilities of all of the B-120VCA alloys were very low due to contamination during processing. Also, the ductility of the Ti-8Al-1Mo-1V alloys was below normal, apparently due to contamination during processing. There was some indication that boron acts as a scavenging agent in titanium, and there were very significant indications that strontium may have beneficial scavenging effect in high-aluminum titanium-base alloys.

Approved for Public Release

Contrails

It is recommended that the elements boron, sulfur, and strontium be further evaluated as grain growth inhibitors in titanium-base alloys. It is also recommended that the scavenging effects observed in this work receive further attention.

Approved for Public Release

Contrails

REFERENCES

1. A. J. Griest, A. P. Young, and P. D. Frost, "Relation between Beta Grain Size and Ductility of High-Strength Alpha-Beta Titanium Alloy," Trans. Met. Soc. AIME, 215 (1959), pp. 844-8.

2. F. A. Crossley and W. F. Carew, "Comparison of Creep-Rupture Properties of Widmanstätten and Equiaxed Structures of Ti-7Al-3Mo Alloys," Trans. AIME, 212 (1958), pp. 748-50.

3. C. J. Sparks, Jr. and J. P. Hammond, "Preferred Orientations and Kinetics of Recrystallization in Titanium," University of Kentucky, WADC TR 56-421, ASTIA AD 110470, July 1956.

4. R. L. Fullman, "Boundary Migration During Grain Growth," Metal Interfaces, ASM, Novelty Park, Ohio (1952), p. 186.

5. W. A. Weyl, "A New Approach to Surface Chemistry and to Heterogeneous Catalysis," Mineral Industries Experiment Station Bulletin No. 57, The Pennsylvania State College (1951), 118 pp.

6. L. N. Berger, D. N. Williams, and R. I. Jaffee, "The Effect of Sulphur on the Properties of Titanium and Titanium Alloys," Trans. ASM, 49 (1957), p. 300.

7. R. Reinbach, "The Recrystallization of Titanium and the Effects of Alloying Additives," Z. Metallkunde, 51 (Jan. 1960), pp. 24-8.

8. J. Winter and S. Weinig, "The Grain Boundary Adsorption of Solutes," Trans. Met. Soc. AIME, 215 (1959), pp. 74-82.

9. Private communication from Thomas Redden, The General Electric Co., Evendale, Ohio.

10. Y. C. Lui, "Grain Size Control in Titanium and the Effect Thereon of Various Addition Agents," Final Report to Watertown Arsenal Laboratory under Contract No. DA-30-069-ORD-840.

11. K. Lucke and K. Detert, "A Quantitative Theory of Grain-Boundary Motion and Recrystallization of Metals in the Presence of Impurities," Acta Metallurgica, 5 (11) (November, 1957), pp. 628-37.

12. F. A. Crossley, Discussion to "Mechanical Properties of Alpha Titanium as Affected by Structures and Composition," Trans. AIME, 203 (1955), pp. 717-18.

13. H. D. Adenstedt, J. R. Pequiquot, and J. M. Raynor, "The Titanium-Vanadium System," Trans. ASM, 44 (1952), p. 990.

14. E. K. Winterfeldt, "Electropolishing with Special Reference to Two Commercial Types of Polishing Apparatus of Metallographic Purposes--Postscript," Mikroskopie, 5 (1950), p. 184.

15. P. Feltham, "Grain Growth in Metals with Special Reference to High-Purity Nickel," Jnl. Inst. Metals, 86 (1957-58), pp. 95-97.

Approved for Public Release

Contrails

TABLE I

SOLUTE CANDIDATES FOR GRAIN GROWTH INHIBITION

Solute	At. No.	Group No.	Ionization Potential, ev*	Goldschmidt Atomic Diameter, kX	Size Factor, %	Melting Point, °F (°C)	Boiling Point, °F (°C)	Quantity to be Added, at. % (wt%)	Solubility in Ti, wt %	
									β at 1800°F	α at 1500°F
Group A - Indicated to be Effective by Experiment										
B	5	III		1.94	-34	3690(2030)	---------	0.5(0.11)	< 0.05	< 0.05
S	16	VI		(1.04)	-65	247(119)	832(444)	0.03(0.05)	~0.012	~0.014
Ag	47	IB		2.88	-1.7	1761(961)	4010(2210)	0.2(0.45)	26	13
Hf	72	IVA		3.17	8.2	4032(2222)	9750(5400)	0.2(0.74)	100	100
Group B - Atoms Anticipated to Have High Polarizability										
Cs	55	I	3.89	5.40	84	83(29)	1273(690)			
Rb	37	I	4.18	5.02	71	102(39)	1270(688)			
Ba	56	II	5.21	4.48	53	1317(714)	2980(1640)	0.2(0.57)		
Sr	38	II	5.69	4.29	46	1414(768)	2520(1380)	0.2(0.36)		
Ti+	22	IV	6.83	2.93		3035(1668)	5900(3260)			

* Electron volts.

\+ For comparison.

Approved for Public Release

Contrails

TABLE II

MATERIALS: SOURCE, FORM, AND PURITY

Element	Purity, %	Supplier	Form	Impurities, wt. %
Aluminum	99.80	Aluminum Co. of America	Granulated	0.10Fe-0.018Ga-0.082Si
Barium	99.0	A. D. McKay, Inc.	Rod	
Boron	99.65	Shieldalloy (Herman C. Starck)	Powder	0.15Fe-0.10Si
Chromium	99.75	Charles Hardy, New York	Powder: 50 mesh with 2% max. on 100	
Chromium	99.43	Elchrome	Lump	0.045C-0.033 O
Hafnium	97	A. D. McKay, Inc.	Iodide	
Molybdenum	99.9	Fansteel Metallurgical Corp.	Chips	0.005Fe, 0.015C, 0.045 O, 0.02 Co + Ni
Silver	99.99	Goldsmith Bros., Div. of National Lead	Granules	
Strontium	99.0	A. D. McKay, Inc.		
Sulfur	99	LaPine Scientific Co.	Lump	
Titanium	(102 Bhn)	E. I. DuPont De Nemours & Co.	Sponge: -7/16 + 0.053 in.	0.04Fe
Vanadium	98.2	Electrometallurgical Div. Union Carbide and Carbon	V-14Al master: -8 mesh	0.5Fe, 0.4Si, 0.03C

Approved for Public Release

Contrails

TABLE III

ROLLING SCHEDULE OF ALLOYS

Alloy Group	Beta Transus Temperature, °F	Rolling Schedule
Unalloyed titanium	1625	From 1 3/4 to 1/2 in. at 1500°F, first 2 passes 0.050 in. and remainder 0.100 in. From 1/2 to 1/4 in. at 1400°F in passes of 0.100 in., and from 1/4 to 0.120 in. at 1300°F in passes of 0.050 in.
B-120VCA	1300 ± 25	From 1 3/4 to 1/4 in. at 1800°F, first 2 passes 0.050 in. and remainder 0.100 in. From 1/4 to 0.120 in. at 1700°F in passes of 0.025 in.
Ti-8Al-1Mo-1V	1900 ± 15	From 1 3/4 to 0.120 in. at 1800°F. First 2 passes at 0.050 in., passes of 0.100 in. to 1/4 in. thickness, and then 0.050 in. passes to 0.120 in.

Approved for Public Release

Contrails

TABLE IV

CHEMICAL ANALYSIS OF ALLOYS*

Alloy No.	Nominal Composition, wt%	Analysis, wt%			
		Al	Cr	Mo	V
B1	13V-11Cr-3Al	3.26	5.53	----	11.7
B3	13V-11Cr-3Al-0.45Ag-0.74Hf	3.21	5.19	----	11.9
C1	8Al-1Mo-1V	7.98	----	0.97	1.08
C5	8Al-1Mo-1V-0.36Sr	7.49	----	0.66	0.89

* Analyses by Charles C. Kawin Co., Chicago, Illinois.

Approved for Public Release

Contrails

TABLE V

GAS ANALYSIS OF TENSILE TEST SPECIMENS*

Specimen No.	Alloy	Gas Analysis, wt%		
		Oxygen	Hydrogen	Nitrogen
A1-1	Unalloyed Ti	0.0732	0.0018	0.0156
B1-3	B-120VCA	0.291	0.0171	0.0030
B3-3	B-120VCA+0.45Ag+0.74Hf	0.209	0.0155	0.0084
C1-5	Ti-8Al-1Mo-1V	0.199	0.0079	0.0213
C5-5	Ti-8Al-1Mo-1V+0.36Sr	0.155	0.0070	0.0028

* Vacuum fusion analyses by Chemical Research Services, Inc., Addison, Illinois.

Approved for Public Release

Contrails

TABLE VI

UNALLOYED TITANIUM GROUP, MEAN GRAIN DIAMETER

$(10^{-3}$ inch)

Specimen No.	Temp., °F	Time, hr	Minor Alloy Addition, wt%					
			None	0.05S	0.45Ag-0.74Hf	0.36Sr	0.11B	Multiple*
2			0.68	0.45	0.90	1.4	0.69	0.86
4	1700	1/4	9.7	a	8.8	----	2.5	9.8
5		1/2	15.8	a	12.7	----	3.7	5.6
6		1	10.9	a	14.9	14.2	3.0	7.6
7		2	16.4	a	20.0	20.0		6.4
8		4	----	a	23.5	23.6	4.4	9.1
9		24	75	a	27.3	50	5.0	----
10	1750	1/4	17.4	1.87	12.5	12.5	2.9	4.4
11		1/2	9.8	4.6	15.0	16.0	3.4	8.8
12		1	12.5	----	13.6	13.0	3.9	5.9
13		2	22.1	3.4	21.4	18.2	4.9	7.1
14		4	18.2	4.6	20	20.0	4.4	7.1
15		24	30	5.3	20	22.2	6.1	----
16	1800	1/4	17.4	2.7	12.5	12.5	2.3	5.8
17		1/2	12.5	---	13.6	16.7	4.3	7.1
18		1	13.6	2.4	18.2	17.4	3.6	4.7
19		2	20.0	2.6	23.5	----	---	9.4
20		4	50	6.6	27.3	20.0	5.6	9.7
21		24						---
22	1850	1/4	12.5	5.2	18.8	16.7	5.1	5.7
23		1/2	14.8	6.2	14.3	14.3	4.5	6.0
24		1	16.7	10.5	27.3	14.3	4.1	8.3
25		2	16.7	10.7	23.1	17.6	5.1	8.6
26		4	36.4	7.0	37.5	23.5	---	8.8
27		24	----	10.5	26.7	27.3	4.6	11.1

* 0.03S-0.12Ba-0.15Hf-0.07Sr-0.01B.

(a) Alpha-beta microstructure; that is, annealed below the beta transus temperature.

Approved for Public Release

Contrails

TABLE VII

B-120VCA (Ti-13V-11Cr-3Al) GROUP MEAN GRAIN DIAMETER
(10^{-3} inch)

Specimen No.	Temp., °F	Time, hr	Minor Alloy Addition, wt%						
			None	0.05S	0.45Ag-0.74Hf	0.57Ba	0.36Sr	0.11B	Multiple*
2			3.5	2.8	4.7	2.1	4.2	2.2	2.2
4	1800	1/4	8.3	5.8	4.4	5.6	5.9	4.2	---
5		1/2	9.8	5.0	8.6	---	----	3.4	4.7
6		1	9.8	---	9.8	5.0	7.6	3.2	5.0
7		2	8.6	5.6	----	---	10.9	3.2	5.0
8		4	10.5	6.2	14.3	5.7	8.9	3.4	8.6
9		24	30.8	6.3	19.0	---	----	---	---
10	1900	1/4	8.5	6.4	10.6	6.0	6.7	3.2	5.0
11		1/2	10.9	6.4	9.4	8.0	7.3	---	5.3
12		1	14.8	7.1	14.2	8.6	7.9	3.2	6.1
13		2	----	6.9	16.4	---	9.8	3.5	---
14		4	22.4	9.4	----	7.1	10.9	---	---
15		24	50.0	7.3	29.5	---	----	---	---
16	2000	1/4	12.0	6.6	----	6.4	10.0	3.6	6.4
17		1/2	11.8	---	----	6.9	10.1	3.8	9.7
18		1	17.2	7.2	20.4	---	15.9	---	----
19		2	24.3	5.9	36.8	---	----	---	10.4
20		4	30.2	9.4	27.4	---	17.2	---	----
21		24	25.1	---	58.4	---	----	---	----
22	2100	1/4	19.0	9.1	10.9	6.7	10.5	4.1	9.8
23		1/2	28.4	7.9	15.6	8.3	14.0	3.8	8.1
24		1	30.0	19.2	37.5	13.9	18.8	4.6	7.3
25		2	33.3	12.3	37.5	12.4	33.6	4.4	8.8
26		4	37.5	8.3	60	12.4	30.0	4.6	9.9
27		24	60.0	25.0	50	----	----	---	---

* 0.03S-0.12Ba-0.15Hf-0.07Sr-0.01B.

Approved for Public Release

Contrails

TABLE VIII

Ti-8Al-1Mo-1V GROUP, MEAN GRAIN DIAMETER
(10^{-3} inch)

Specimen No.	Temp., °F	Time hr	Minor Alloy Addition, wt%						
			None	0.05S	0.45Ag-0.74Hf	0.57Ba	0.36Sr	0.11B	Multiple*
2			0.20	0.19	0.20	0.38	0.25	0.14	0.17
4	1900	1/4	a	3.4	5.0	a	a	2.5	----
5		1/2	a	2.9	8.1	a	a	----	----
6		1	a	----	----	a	a	----	----
7		2	a	----	----	a	a	----	----
8		4	a	----	----	a	a	----	----
9		24	a	----	----	a	a	----	----
10	1950	1/4	----	2.9	6.7	----	5.8	3.0	5.4
11		1/2	----	----	12.4	----	----	3.1	----
12		1	7.3	3.3	-----	----	8.1	----	----
13		2	----	5.4	28.0	----	8.8	4.0	6.6
14		4	----	9.8	-----	----	----	----	----
15		24	----	----	33.6	----	----	----	----
16	2000	1/4	-----	3.3	6.2	-----	10.8	----	----
17		1/2	10.3	3.8	9.9	15.8	-----	3.4	----
18		1	16.0	4.3	16.6	-----	-----	----	----
19		2	27.0	-----	32.9	-----	16.6	----	----
20		4	-----	11.1	33.6	-----	-----	----	----
21		24	-----	----	----	-----	-----	----	----
22	2050	1/4	15.4	3.8	6.4	13.6	16.0	3.0	6.4
23		1/2	14.4	4.2	10.9	10.9	14.6	4.0	5.3
24		1	16.2	5.3	14.0	12.9	19.8	3.0	5.5
25		2	19.6	5.0	18.7	----	18.7	2.8	5.9
26		4	27.0	7.4	16.8	21.8	19.8	4.7	8.0
27		24	-----	----	----	----	-----	----	----

* 0.03S-0.12Ba-0.15Hf-0.07Sr-0.01B.

[a] Alpha-beta microstructure; that is, annealed below beta transus temperature.

Approved for Public Release

Contrails

TABLE IX

"STABILIZED" MATERIALS, MEAN GRAIN DIAMETER

(10^{-3} inch)

Time, hr	Temp., °F	Ti-base		Ti-0.05S		Temp., °F	B-120VCA-base		B-120VCA-0.05S	
		Stabilized	Standard*	Stabilized	Standard		Stabilized	Standard*	Stabilized	Standard
Initial		0.37	0.68	0.33	0.45		8.1+	3.5	5.0+	2.8
1/4	1700	12.2	9.7	a	a	1800	5.3	8.3	3.0	5.8
1/2		12.0	15.8	a	a		7.2	9.8	5.2	5.0
1		10.2	10.9	a	a		6.9	9.8	3.4	---
2		15.1	16.4	a	a		12.1	8.6	6.0	5.6
4		20.2	----	a	a		11.2	10.5	5.0	6.2
1/4	1750	12.1	17.4	4.4	1.87	1900	8.3	8.5	4.3	6.4
1/2		15.8	9.8	11.2	4.6		7.9	10.9	7.0	6.4
1		14.8	12.5	----	----		12.1	14.8	10.6	7.1
2		18.6	22.1	9.6	3.4		14.8	----	4.5	6.9
4		21.4	18.2	10.7	4.0		15.4	22.4	4.8	9.4
1/4	1800	8.6	17.4	12.2	2.7	2000	8.4	12.0	6.2	6.6
1/2		16.1	12.5	8.8	---		15.7	11.8	5.1	---
1		20.0	16.9	11.1	2.4		23.6	17.2	8.6	7.2
2		20.2	20.0	16.9	2.6		19.7	24.3	5.4	5.9
4		45.4	50	18.6	6.6		24.8	30.2	8.2	9.4
1/4	1850	11.0	12.5	10.7	5.2	2100	14.3	19.0	6.6	9.1
1/2		20.2	14.8	7.9	6.2		20.5	28.4	6.3	7.9
1		26.9	16.7	14.8	10.5		23.6	30.0	8.4	19.2
2		20.2	16.7	14.8	7.0		39.3	33.3	11.0	12.3
4		24.2	36.4	23.6	10.5		----	37.5	6.7	8.3

* Standard data taken from Tables VI and VII.

\+ Stress-relieved but not recrystallized.

a Alpha-beta microstructures; that is, annealed below the beta transus temperature.

Approved for Public Release

Contrails

TABLE X

TENSILE TEST PROPERTIES OF UNALLOYED TITANIUM GROUP WITH MINOR ADDITIONS

Specimen No.	Addition, wt%	Yield Strength (0.2% Offset), ksi	Ultimate Tensile Strength, ksi	Elongation, %
		Annealed: 1300°F-1 hr-AC		
A1-1	None	50.0	65.8	25.7
A1-2		49.2	65.8	23.3
		49.6 av.	65.8 av.	24.5 av.
A2-1	0.05S	49.5	66.0	24.9
A2-2		49.6	65.0	27.9
		49.6 av.	65.5 av.	26.4 av.
A3-1	0.45Ag-0.74Hf	48.9	65.1	29.2
A3-2		43.6	64.9	25.0
		46.2 av.	65.0 av.	27.1 av.
A5-1	0.36Sr	43.1	62.8	27.6
A5-2		43.1	62.5	22.6
		43.1 av.	62.6 av.	25.1 av.
A6-1	0.11B	45.0	58.0	28
A6-2		42.0	58.2	27.6
		43.5 av.	58.1 av.	27.8 av.
A7-1	0.03S-0.12Ba-0.15Hf-0.07Sr-0.01B	37.1	54.3	26.5
A7-2		38.2	55.1	26.1
		37.6 av.	54.7 av.	26.3

Approved for Public Release

Contrails

TABLE XI

TENSILE TEST PROPERTIES OF B-120VCA (Ti-13V-11Cr-3Al) GROUP WITH MINOR ADDITIONS

Specimen No.	Addition, wt%	Yield Strength (0.2% Offset), ksi	Ultimate Tensile Strength, ksi	Elongation, %
	Solution Heat Treated: 1450°F-20 min-AC			
B1-1	None	95.2	97.0	1.3
B1-2		95.6	98.4	1.3
B2-1	0.05S	93.2	95.0	.59
B2-2		82.2	84.2	.55
B3-1	0.45Ag-0.74Hf	121.0	123.0	2.2
B3-2		121.0	121.6	2.8
B4-1	0.57Ba	-----	79.6	----
B4-2		-----	58.6	----
B5-1	0.36Sr	-----	114.0	----
B5-2		-----	110.0	----
B6-1	0.11B	-----	120.6	----
B6-2		-----	118.6	----
B7-1	0.03S-0.12Ba-0.15Hf	131.8	132.4	2.7
B7-2	0.07Sr-0.01B	134.0	134.4	3.6
	Solution Heat Treated and Aged: 1450°F-20 min-AC, 900°F-40 hr-AC			
B1-3	None	-----	48.8	----
B1-4		-----	66.0	----
B2-3	0.05S	-----	70.8	----
B2-4		-----	79.6	----
B3-3	0.45Ag-0.74Hf	-----	162.0	----
B3-4			158.0	----
B4-3	0.57Ba	-----	69.6	----
B4-4		-----	30.7	----
B5-3	0.36Sr	-----	97.6	----
B5-4		-----	111.4	----
B6-3	0.11B	-----	114.4	----
B6-4		-----	97.6	----
B7-3	0.03S-0.12Ba-0.15Hf	-----	152.8	----
B7-4	0.07Sr-0.01B	-----	121.6	----

Approved for Public Release

Contrails

TABLE XII

TENSILE TEST PROPERTIES OF Ti-8Al-1Mo-1V GROUP WITH MINOR ADDITIONS

Specimen No.	Addition, wt%	Yield Strength (0.2% Offset), ksi	Ultimate Tensile Strength, ksi	Elongation, %
	Annealed: 1400°F-24 hr-AC			
C1-1	None	155.6	157.6	0.76
C1-2		159.6	160.0	1.8
C2-1	0.05S	136.0	144.0	9.6
C2-2		138.0	156.0	7.9
C3-1	0.45Ag-0.74Hf	117.4	117.4	7.6
C3-2		122.6	124.0	3.7
C4-1	0.57Ba	135.6	137.0	9.6
C4-2		134.0	135.8	11.4
C5-1	0.36Sr	119.6	120.4	12.8
C5-2		118.4	120.8	12.1
C6-1	0.11B	131.2	132.0	9.6
C6-2		130.6	132.0	8.5
C7-1	0.03S-0.12Ba-0.15Hf	140.0	145.6	3.1
C7-2	0.07Sr-0.01B	141.6	147.0	8.7
	Solution Heat Treated: 1950°F-15 min-AC			
C1-3	None	146.2	146.0	0.25
C1-4		147.4	147.4	0.22
C2-3	0.05S	128.4	138.0	1.7
C2-4		127.6	134.0	0.86
C3-3	0.45Ag-0.74Hf	124.6	130.2	2.7
C3-4		120.0	128.2	1.7
C4-3	0.57Ba	133.4	139.6	1.3
C4-4		131.4	139.6	0.88
C5-3	0.36Sr	127.4	137.0	2.9
C5-4		127.6	136.0	2.8
C6-3	0.11B	118.6	119.0	0.30
C6-4		123.8	132.0	1.6
C7-3	0.03S-0.12Ba-0.15Hf	127.8	134.2	1.0
C7-4	0.07Sr-0.01B	125.6	132.2	1.2

Approved for Public Release

Contrails

TABLE XII (Cont.)

Specimen No.	Addition, wt%	Yield Strength (0.2% Offset), ksi	Ultimate Tensile Strength, ksi	Elongation, %
Solution Heat Treated and Aged: 1950°F-15 min-AC, 1100°F-8 hr-AC				
C1-5	None	-----	131.6	----
C1-6		-----	140.4	----
C2-5	0.05S	-----	132.2	----
C2-6		-----	124.0	----
C3-5	0.45Ag-0.74Hf	128.6	129.0	0.34
C3-6		-----	117.6	----
C4-5	0.57Ba	132.0	132.0	0.21
C4-6		128.8	128.8	0.21
C5-5	0.36Sr	131.6	134.0	0.72
C5-6		130.4	134.0	0.60
C6-5	0.11B	-----	124.4	----
C6-6		-----	91.2	----
C7-5	0.03S-0.12Ba-0.15Hf	-----	97.2	----
C7-6	0.07Sr-0.01B	-----	100.4	----

Approved for Public Release

Contrails

TABLE XIII

EVALUATION OF SURFACE CONTAMINATION IN Ti-8Al-1Mo-1V BY MICROHARDNESS MEASUREMENTS OF TENSILE TEST SPECIMENS

Specimen No.	Minor Addition	Heat Treatment	Microhardness (25g Load) DPH 1 mil from surface	 5 mils from surface	 Center
C1-1	None	1400°F-24hr-AC	438	416	433
C1-5	None	1950°F-15min-AC, 1100°F-8hr-AC	485	427	455
C4-1	0.57Ba	1400°F-24hr-AC	400	405	416
C4-5	0.57Ba	1950°F-15min-AC	662	433	416

Approved for Public Release

Contrails

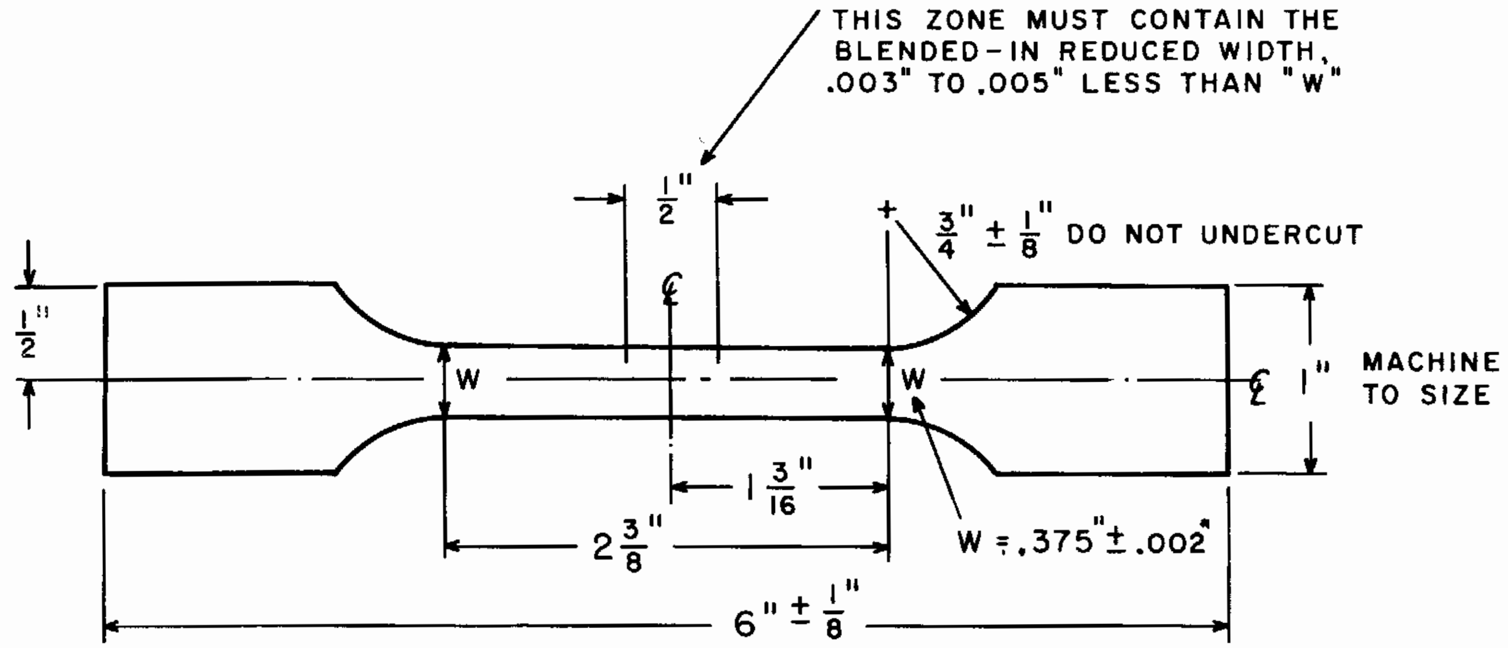
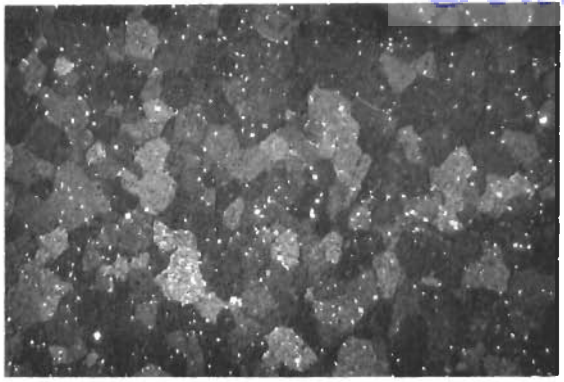


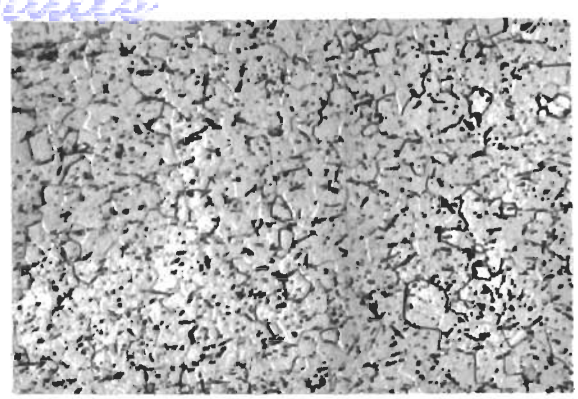
FIG 1 - GEOMETRY OF TENSILE TEST SPECIMEN

Approved for Public Release

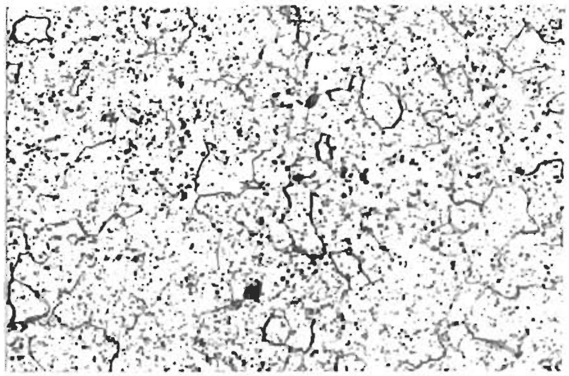
Contrails



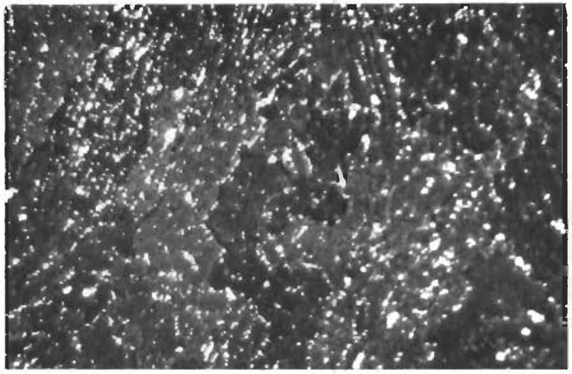
Neg. No. 22769
(a) No addition, polarized light
grain dia. = 0.68 x 10^{-3}in.



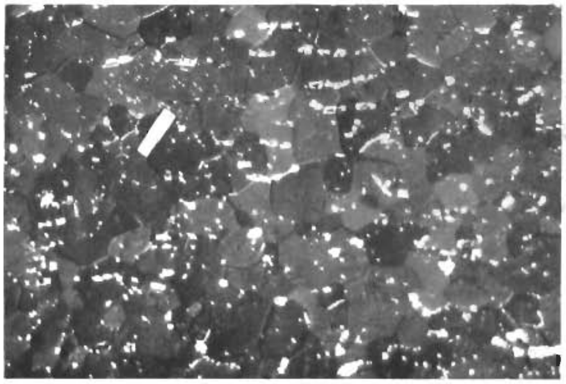
Neg. No. 22770
(b) 0.05S, oblique lighting
grain dia. = 0.45 x 10^{-3}in.



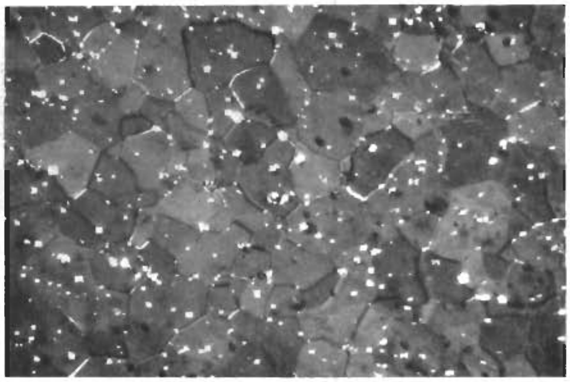
Neg. No. 22772
(c) 0.45Ag-0.7Hf
grain dia. = 0.90 x 10^{-3}in.



Neg. No. 22773
0.36Sr, polarized light
grain dia. = 1.4 x 10^{-3}in.



Neg. No. 22774
(e) 0.11B, polarized light
grain dia. = 0.69 x 10^{-3}in.



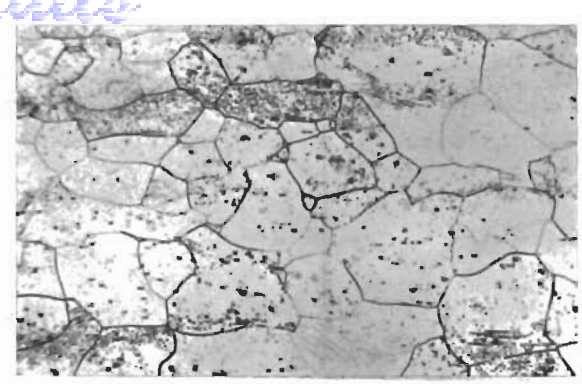
Neg. No. 22775
(f) 0.03S-0.12Ba-0.15Hf-0.07Sr-0.01B, polarized light
grain dia. = 0.86 x 10^{-3} in.

Fig. 2 - Initial microsturctures of unalloyed titanium group, 1300°F-1hr. X250. Etchant: 20% conc. HF, 20% conc, HNO_3, balance glycerine.

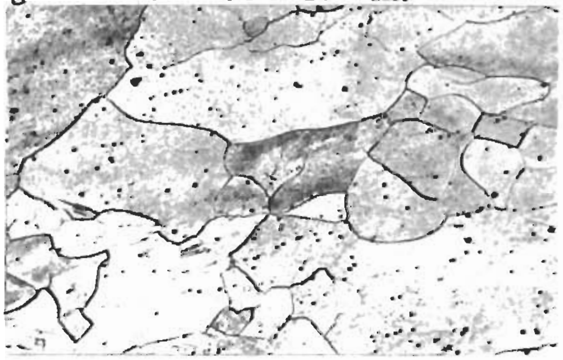
Approved for Public Release

Contrails

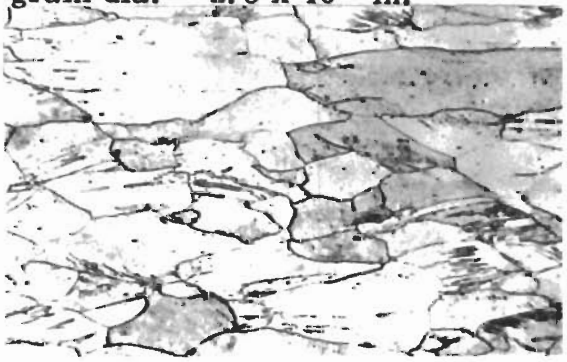
Neg. No. 22777
(a) No addition
grain dia. = 3.5 x 10^{-3}in.



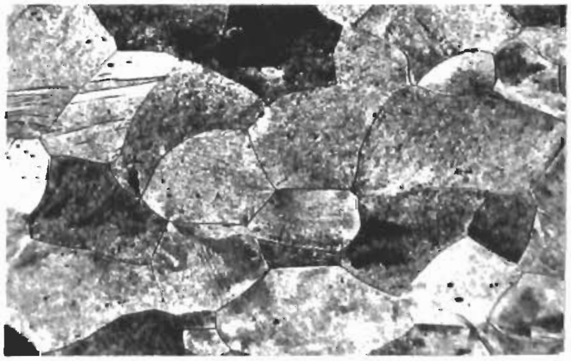
Neg. No. 22778
(b) 0.05S
grain dia. = 2.8 x 10^{-3}in.



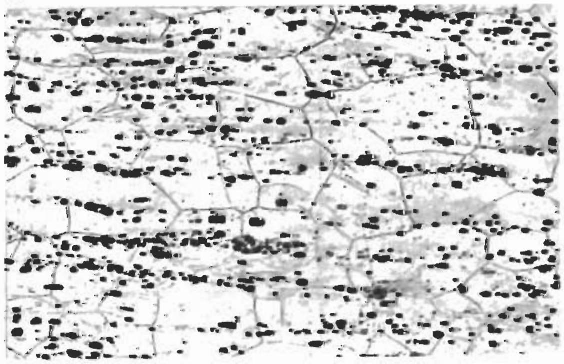
Neg. No. 22779
(c) 0.45Ag-0.74Hf
grain dia. = 4.7 x 10^{-3}in.



Neg. No. 22780
(d) 0.57Ba
grain dia. = 2.1 x 10^{-3}in.



Neg. No. 22781
(e) 0.36Sr
grain dia. = 4.2 x 10^{-3}in.



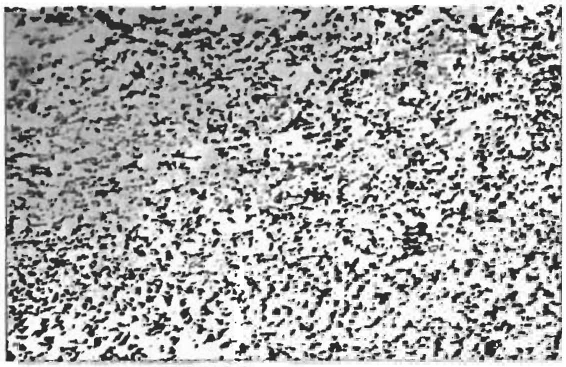
Neg. No. 22782
(f) 0.11B
grain dia. = 2.2 x 10^{-3}in.

Neg. No. 22783
(g) 0.03S-0.12Ba-0.15Hf-0.07Sr-0.01B
grain dia. = 2.2 x 10-3in.

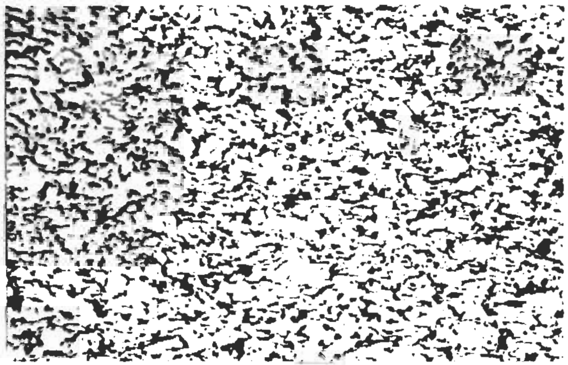
Fig. 3 - Initial microstructures of the B-120VCA group, 1300°F-1hr. X100. Etchant: 20% conc. HF, 20% conc. HNO_3, balance glycerine.

Approved for Public Release

Contrails

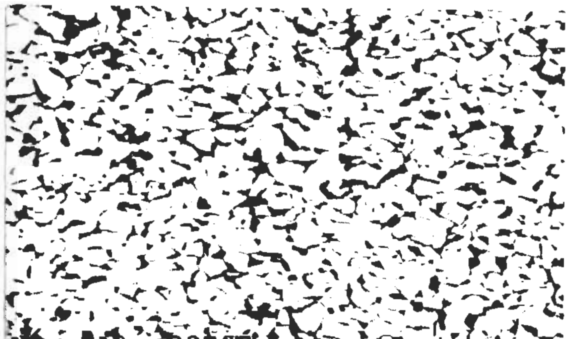


Neg. No. 22784
(a) No addition
grain dia. = 0.20 x 10^{-3}in.

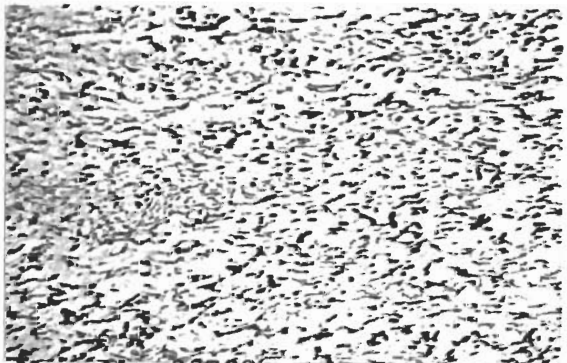


Neg. No. 22785
(b) 0.05S
grain dia. = 0.19 x 10^{-3}in.

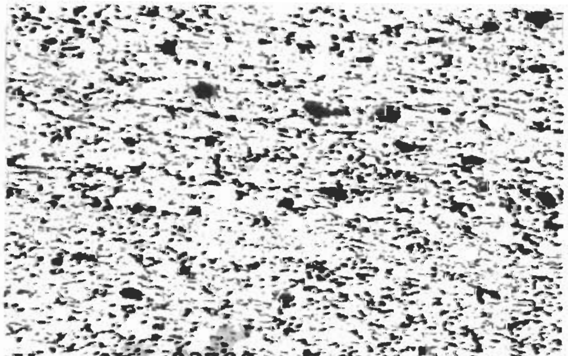
Neg. No. 22786
(c) 0.45Ag-0.74Hf
grain dia. = 0.20 x 10^{-3}in.



Neg. No. 22787
(d) 0.57Ba
grain dia. = 0.38 x 10^{-3}in.



Neg. No. 22788
(e) 0.36Sr
grain dia. = 0.25 x 10^{-3}in.



Neg. No. 22789
(f) 0.11B
grain dia. = 0.14 x 10^{-3}in.

Neg. No. 22790
(g) 0.03S-0.12Ba-0.15Hf-0.07Sr-0.01B
grain dia. = 0.17 x 10^{-3}in.

Fig. 4 - Initial microstructures of Ti-8Al-1Mo-1V group, 1300°F-2hr. X250. Etchant: 20% conc. HF, 20% conc. HNO_3, balance glycerine.

Approved for Public Release

Contrails

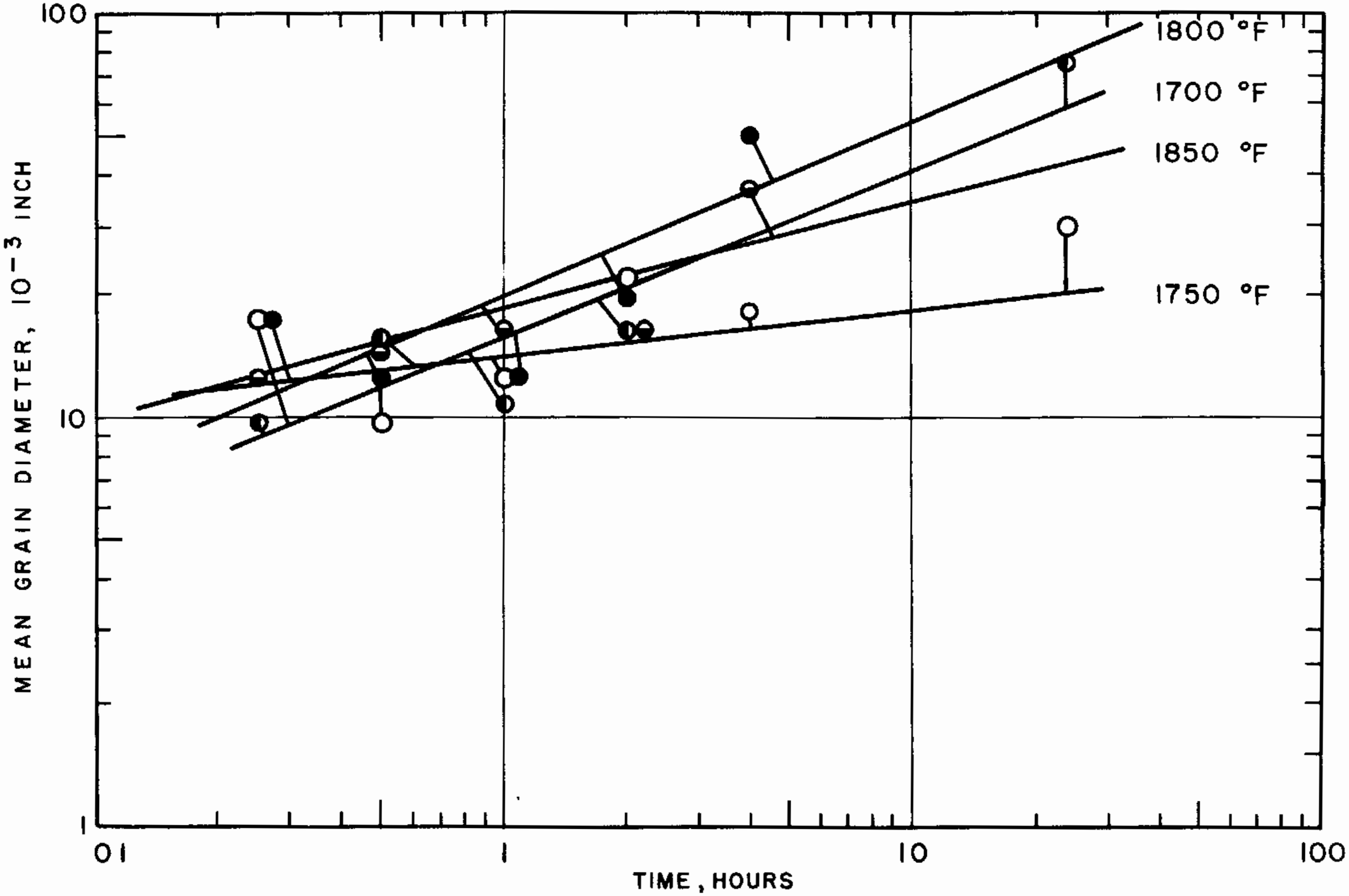


FIG. 5 - RELATIONSHIP OF MEAN GRAIN SIZE OF UNALLOYED TITANIUM WITH ANNEALING TIME.

Approved for Public Release

Contrails

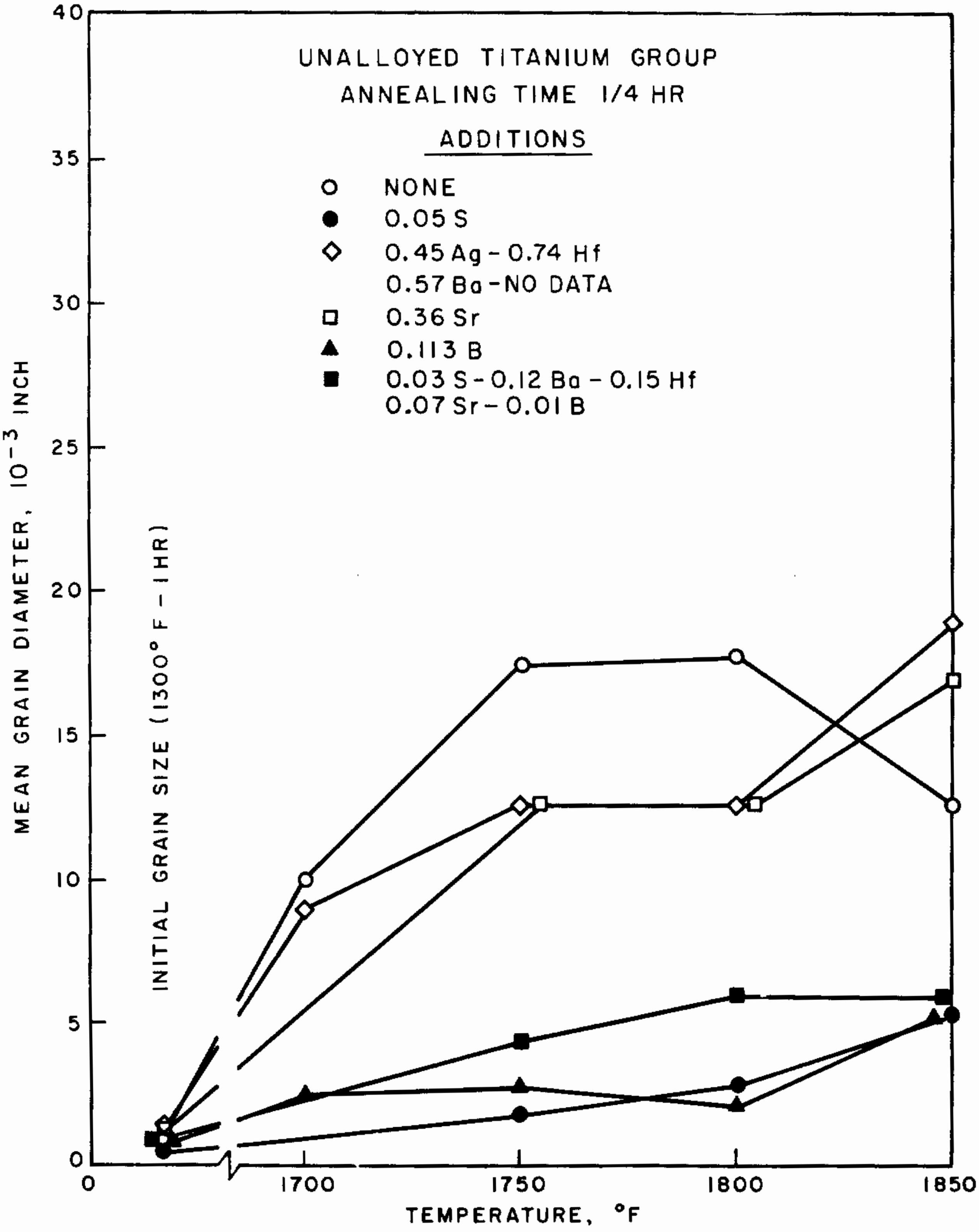


FIG. 6 - COMPARISON OF GRAIN GROWTH OF UNALLOYED TITANIUM GROUP ALLOYS FOR 1/4-HOUR ANNEALS.

Approved for Public Release

Contrails

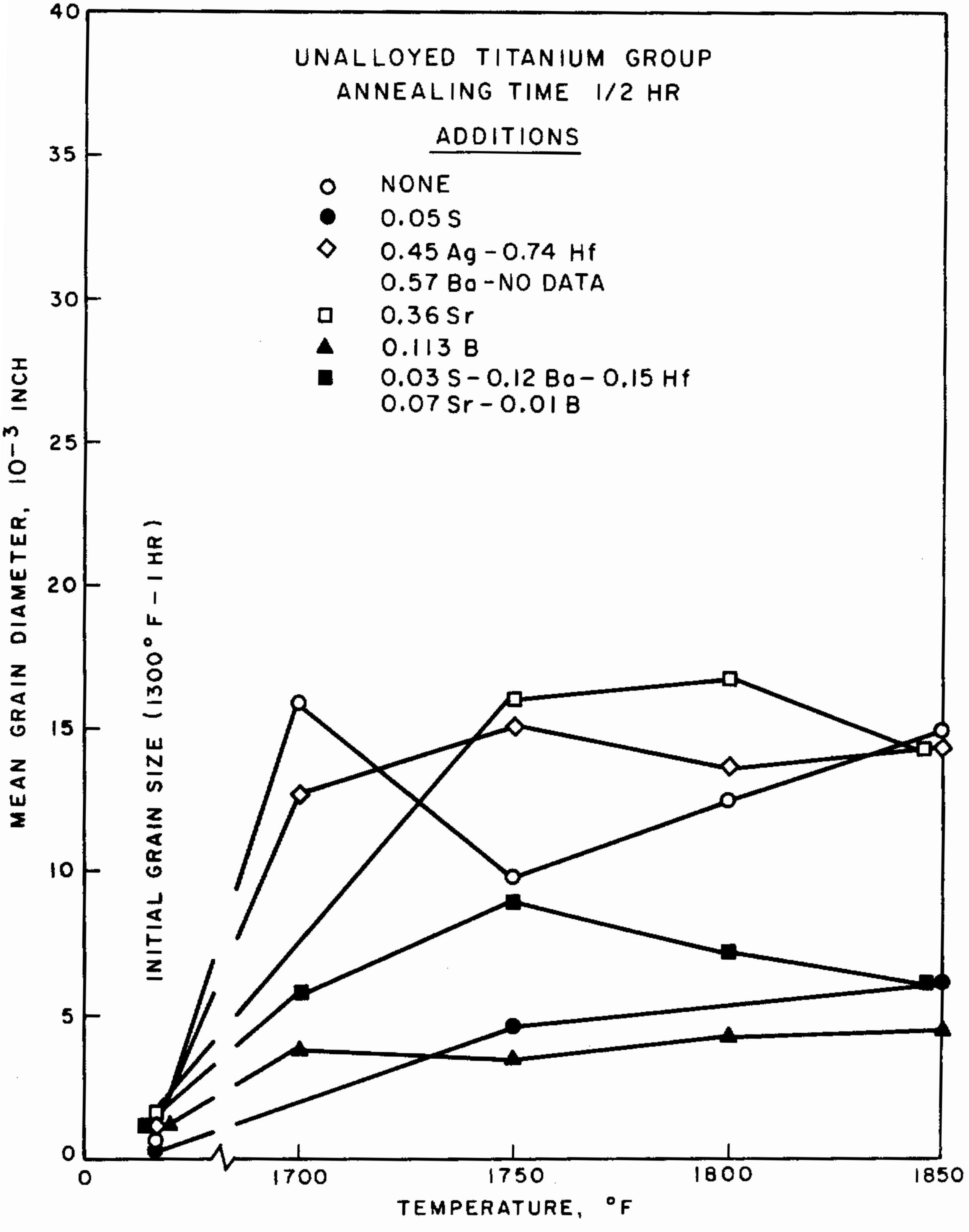


FIG. 7 - COMPARISON OF GRAIN GROWTH OF UNALLOYED TITANIUM GROUP ALLOYS FOR 1/2-HOUR ANNEALS.

Approved for Public Release

Contrails

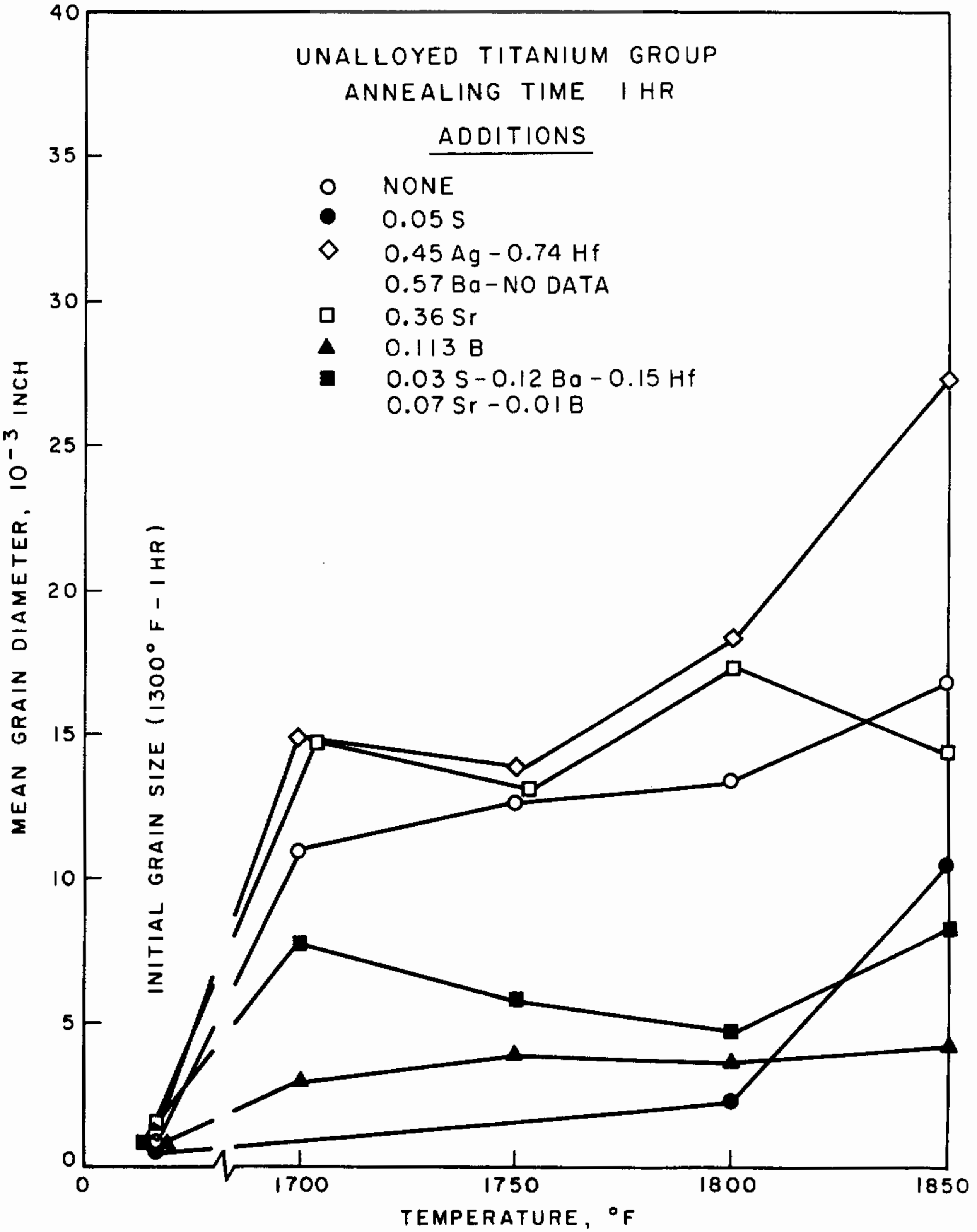


FIG. 8 - COMPARISON OF GRAIN GROWTH OF UNALLOYED TITANIUM GROUP ALLOYS FOR 1-HOUR ANNEALS.

Approved for Public Release

Contrails

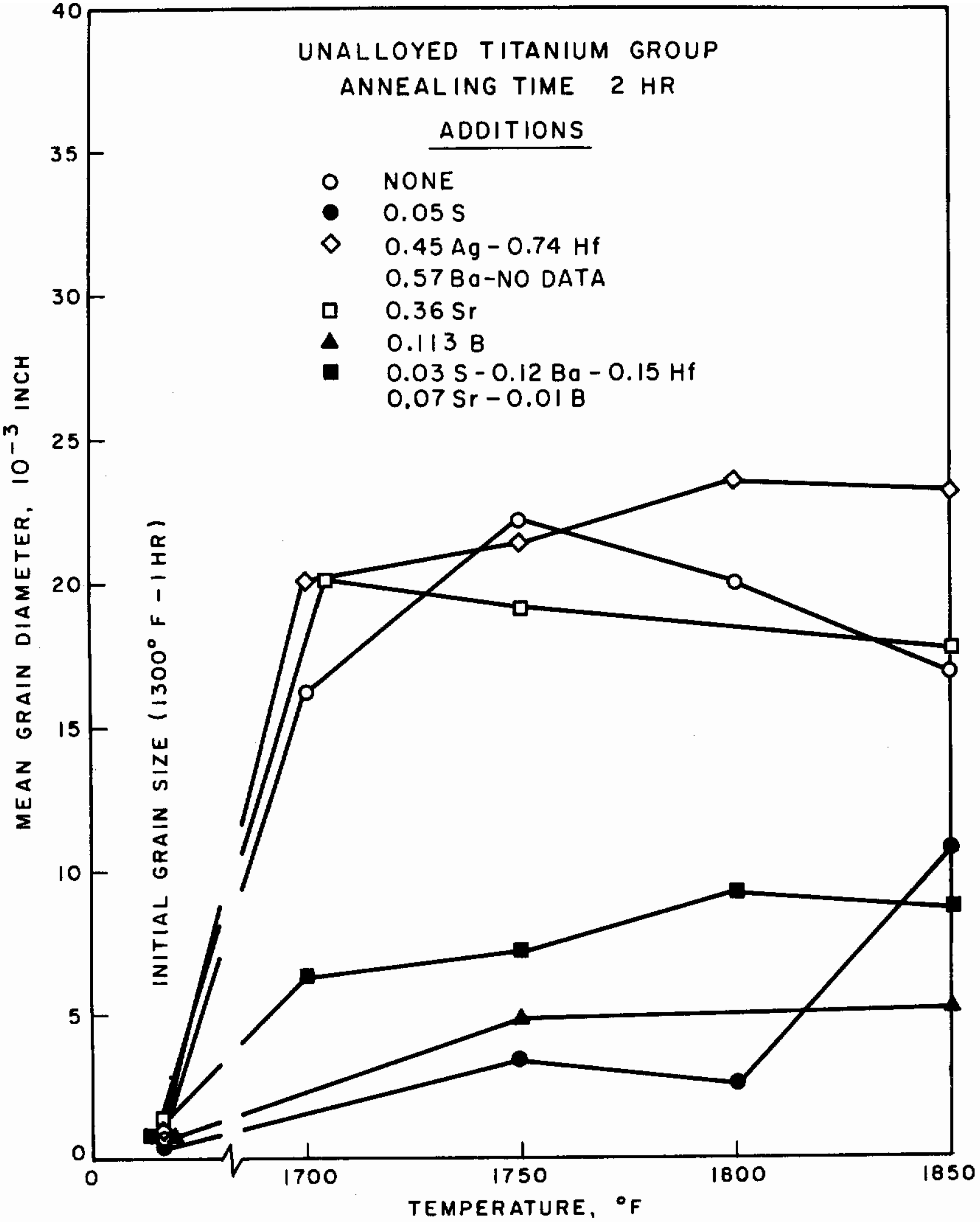


FIG. 9 - COMPARISON OF GRAIN GROWTH OF UNALLOYED TITANIUM GROUP ALLOYS FOR 2-HOUR ANNEALS.

Approved for Public Release

Contrails

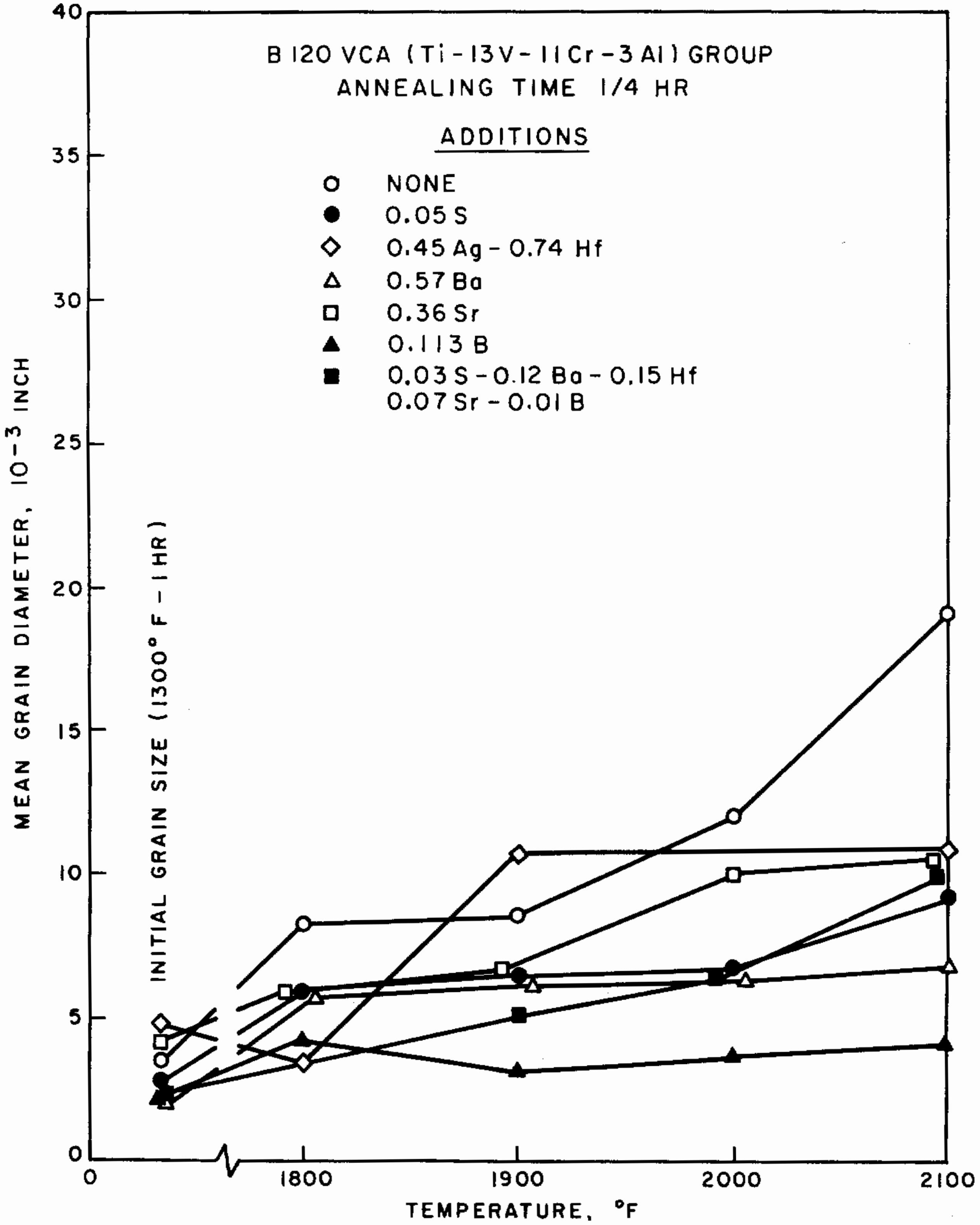


FIG. 10 - COMPARISON OF GRAIN GROWTH OF B-120VCA GROUP ALLOYS FOR 1/4-HOUR ANNEALS.

Approved for Public Release

Contrails

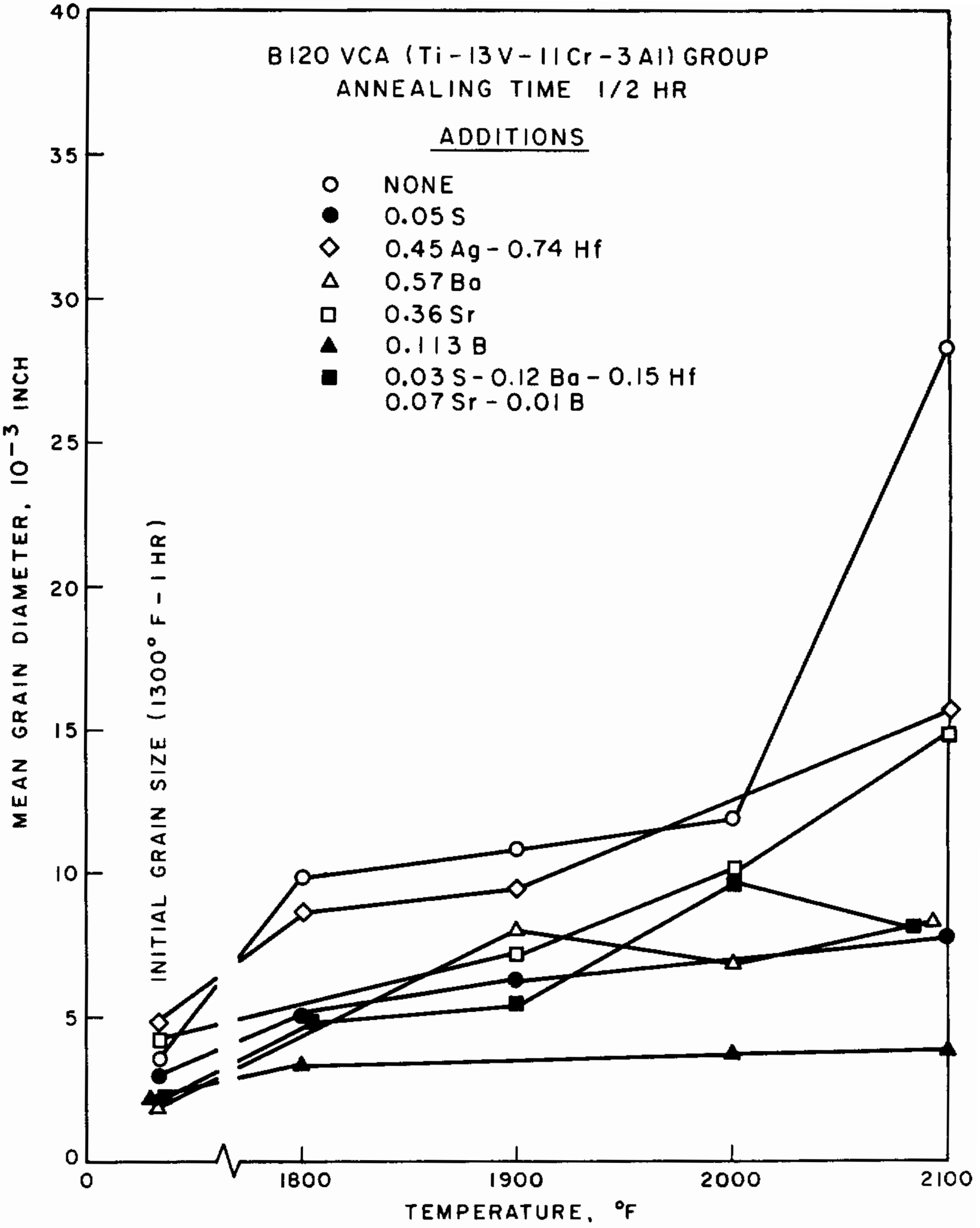


FIG. 11 - COMPARISON OF GRAIN GROWTH OF B-120VCA GROUP ALLOYS FOR 1/2-HOUR ANNEALS.

Approved for Public Release

Contrails

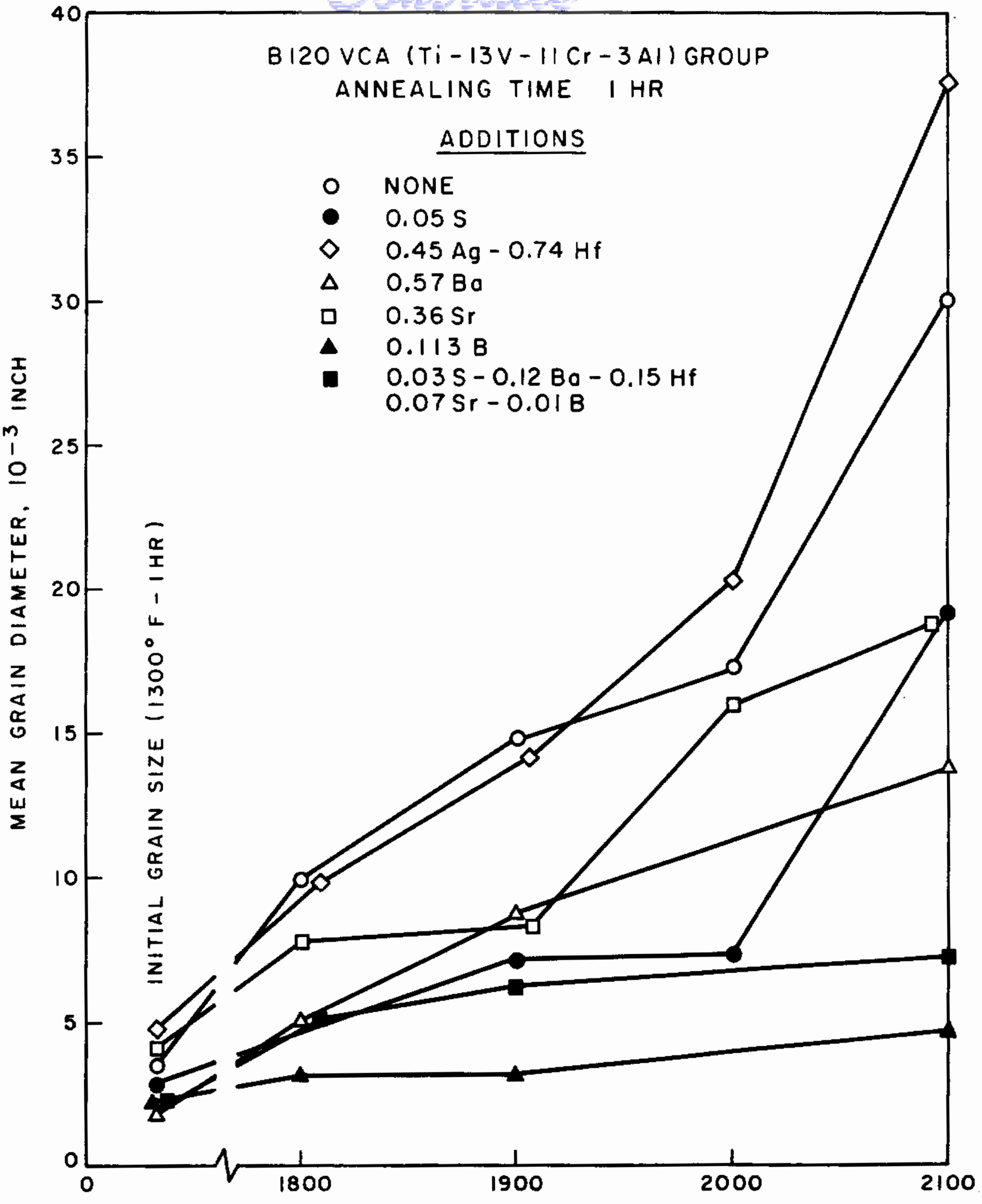


FIG. 12 - COMPARISON OF GRAIN GROWTH OF B-120VCA GROUP ALLOYS FOR 1-HOUR ANNEALS.

Approved for Public Release

Contrails

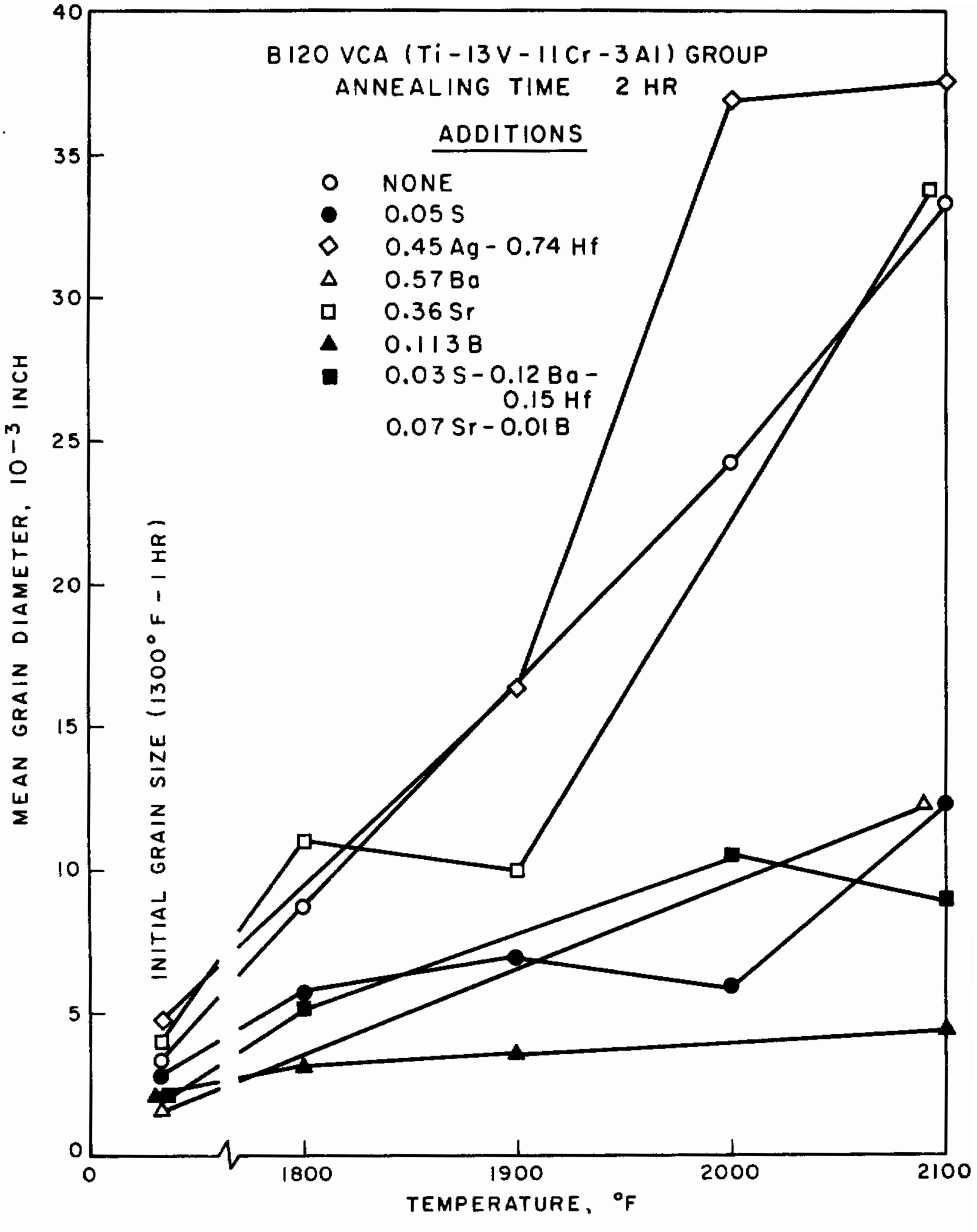


FIG 13 - COMPARISON OF GRAIN GROWTH OF B-120VCA GROUP ALLOYS FOR 2-HOUR ANNEALS.

Approved for Public Release

Contrails

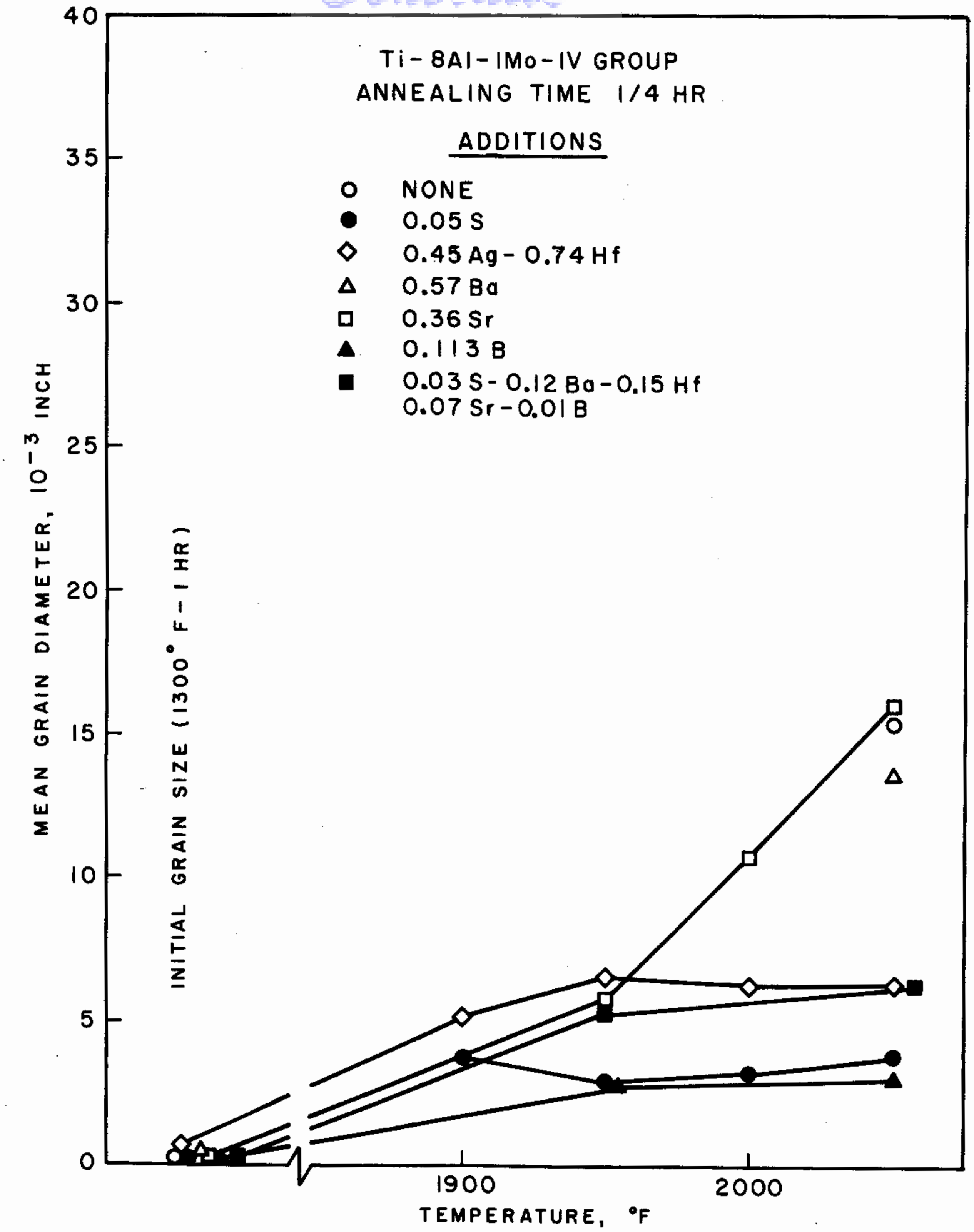


FIG. 14 - COMPARISON OF GRAIN GROWTH OF Ti-8Al-1Mo-1V GROUP ALLOYS FOR 1/4-HOUR ANNEALS.

Approved for Public Release

Contrails

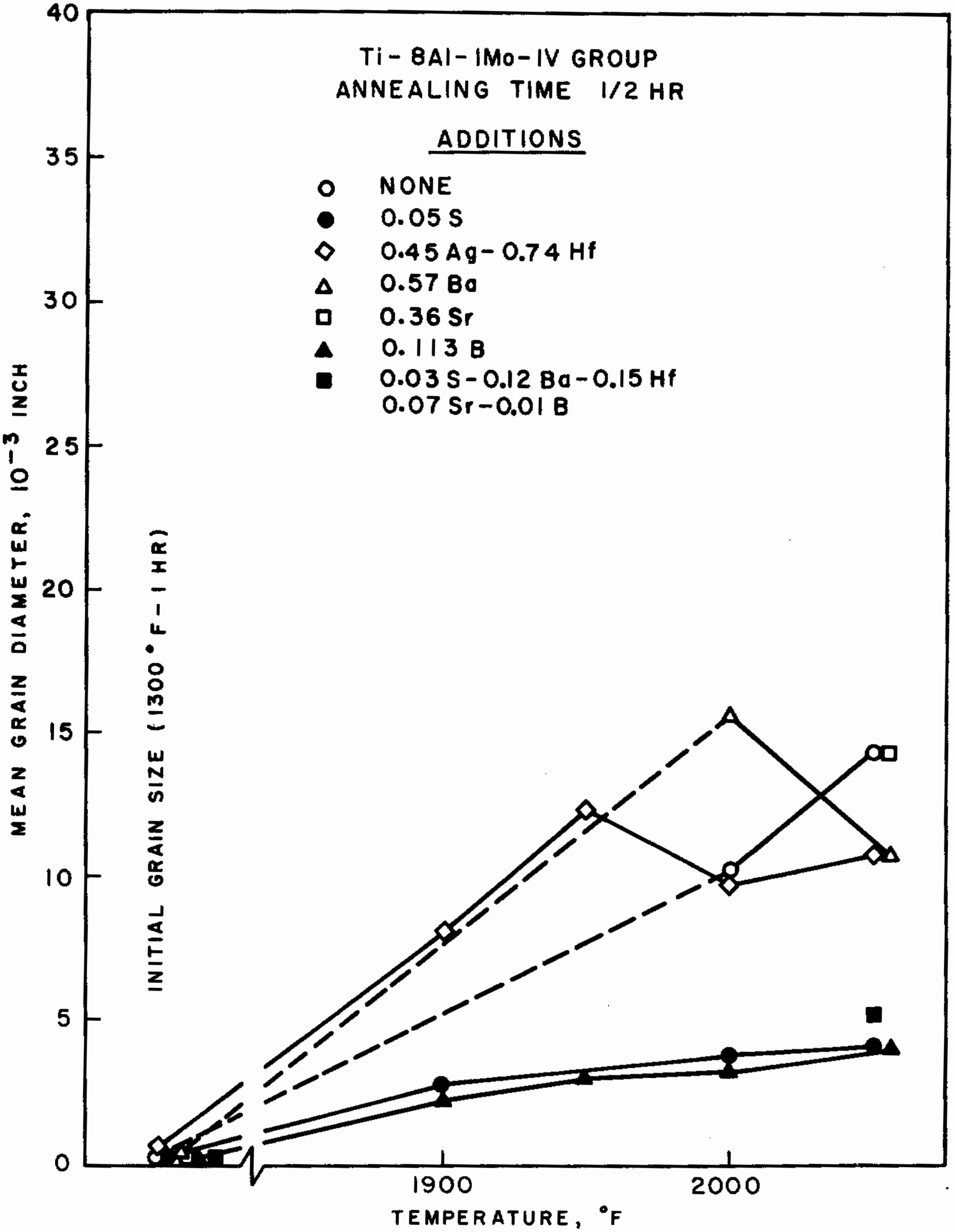


FIG 15 - COMPARISON OF GRAIN GROWTH OF Ti-8Al-1Mo-1V GROUP ALLOYS FOR 1/2-HOUR ANNEALS.

Approved for Public Release

Contrails

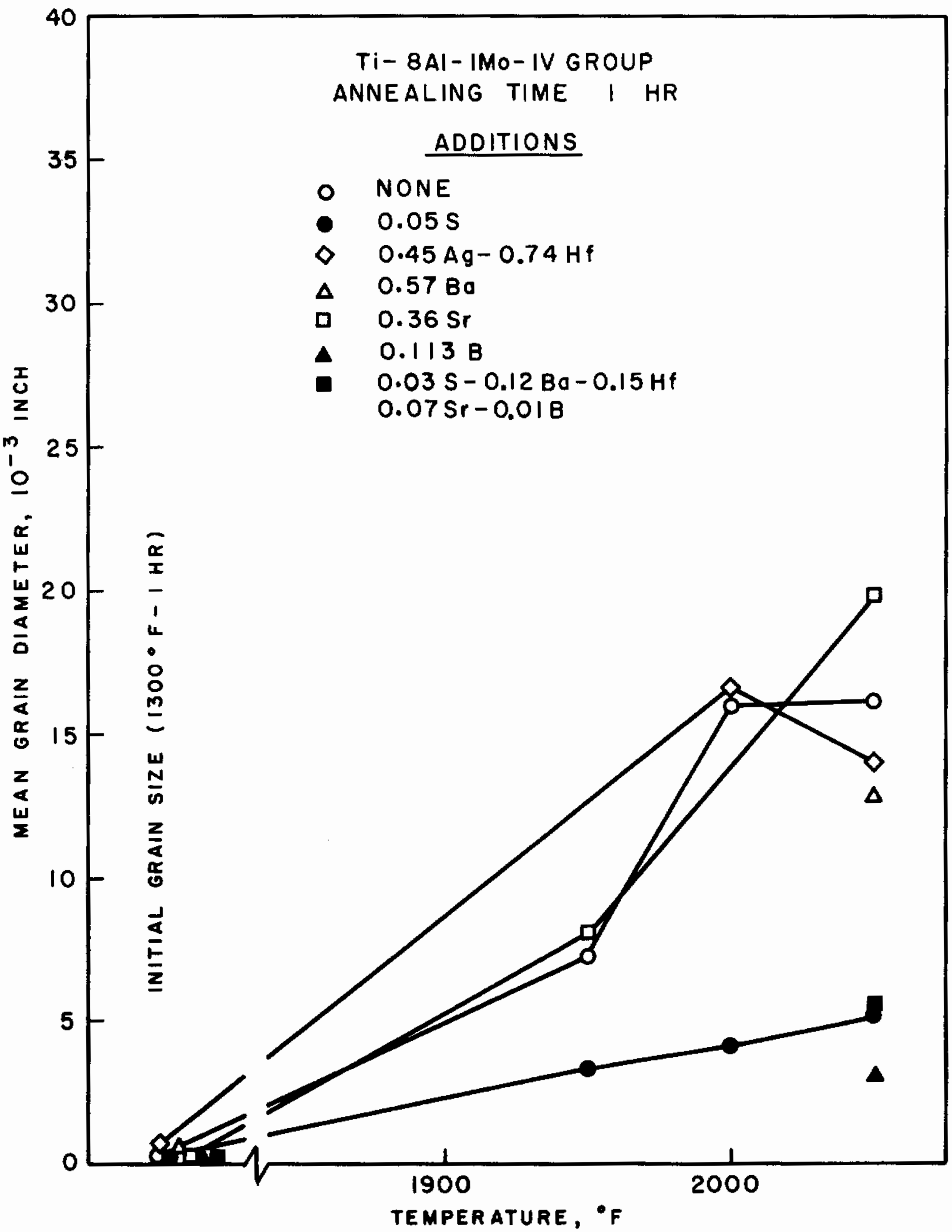


FIG. 16 - COMPARISON OF GRAIN GROWTH OF Ti-8Al-1Mo-1V GROUP ALLOYS FOR 1-HOUR ANNEALS.

Approved for Public Release

Contrails

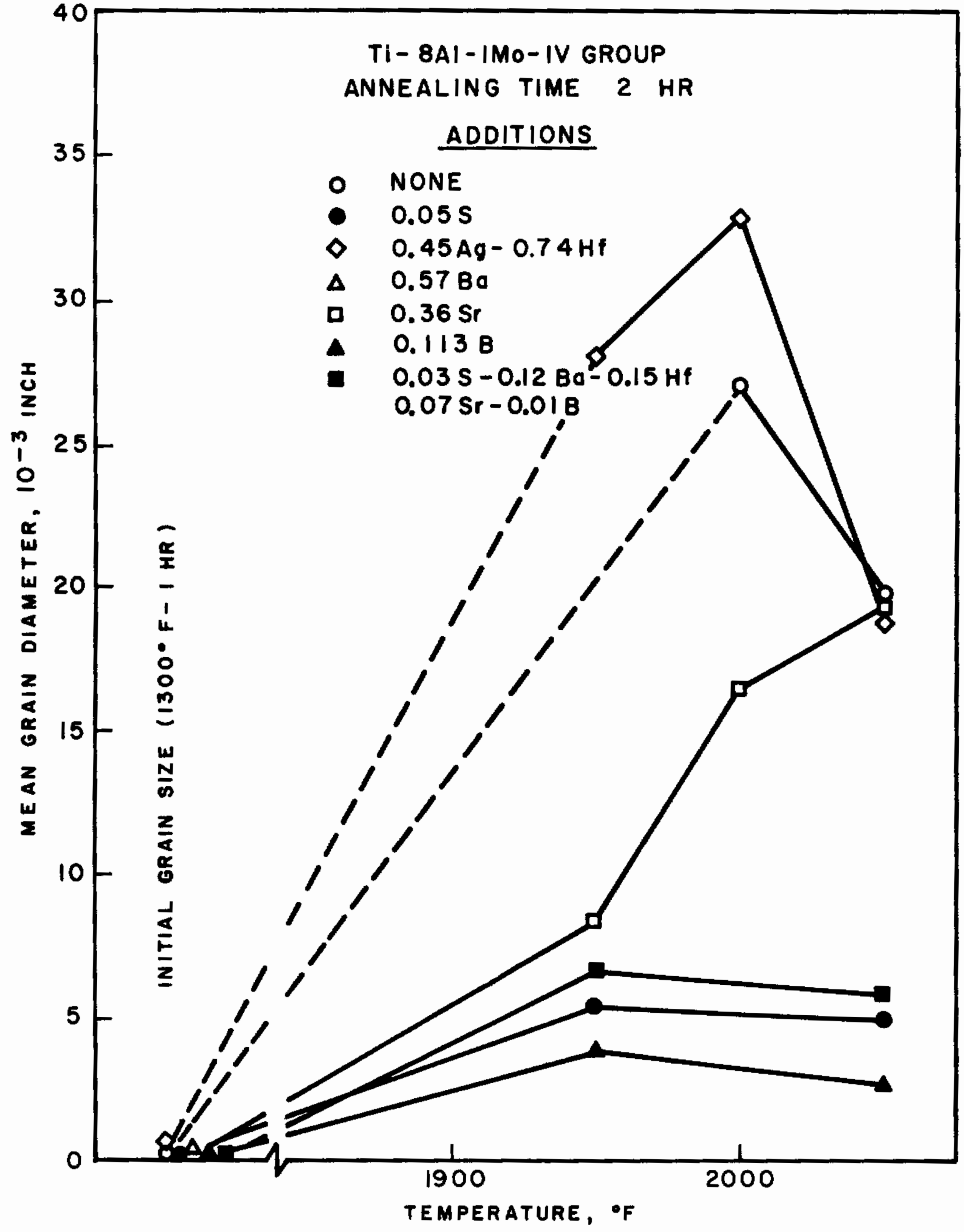


FIG. 17 - COMPARISON OF GRAIN GROWTH OF Ti-8Al-1Mo-1V GROUP ALLOYS FOR 2-HOUR ANNEALS.

Approved for Public Release

Contrails

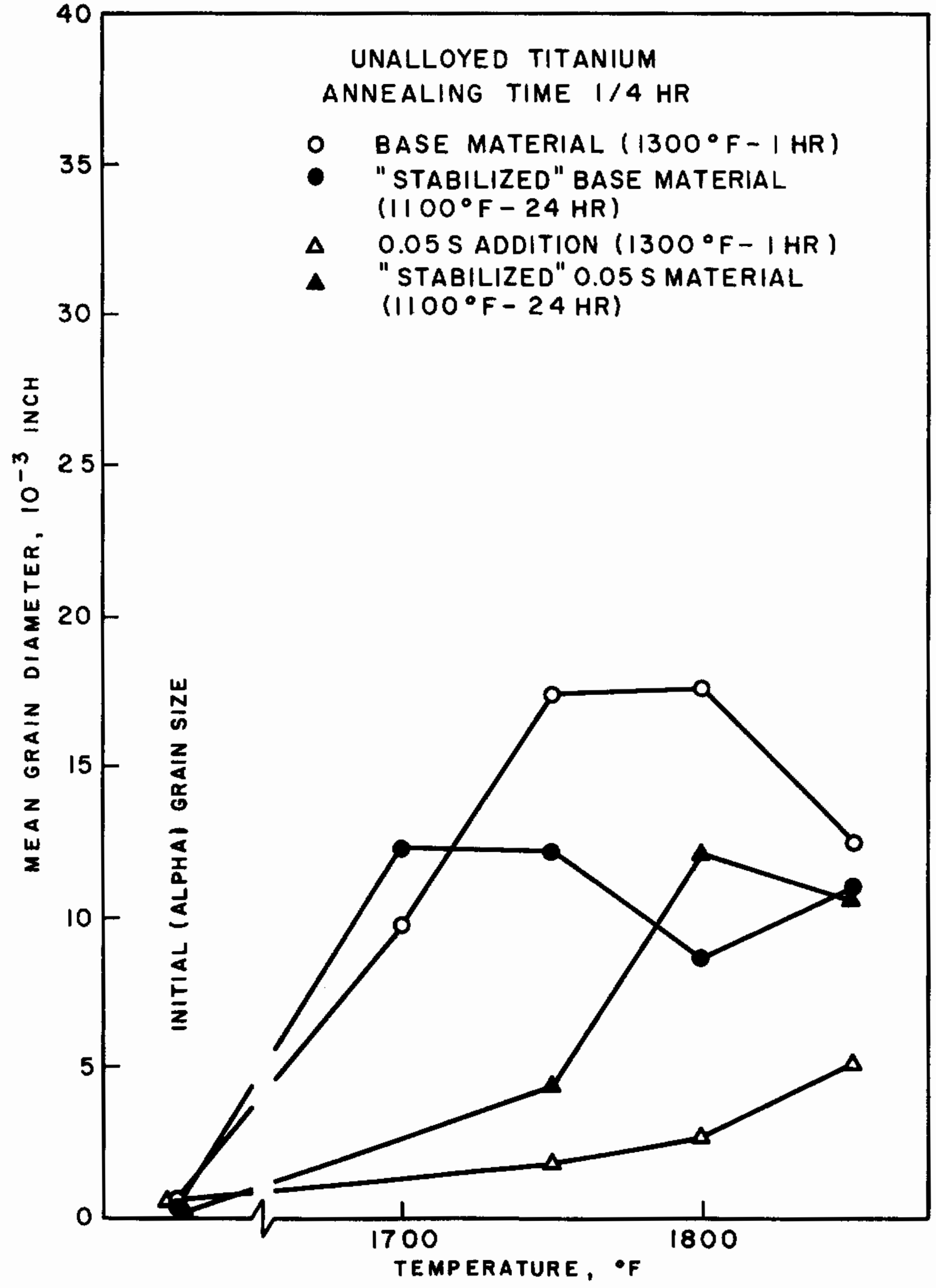


FIG. 18 - COMPARISON OF GRAIN GROWTH OF "STABILIZED" AND STANDARD CONDITIONS OF Ti-BASE AND Ti-0.05S ALLOY FOR 1/4-HOUR ANNEALS.

Approved for Public Release

Contrails

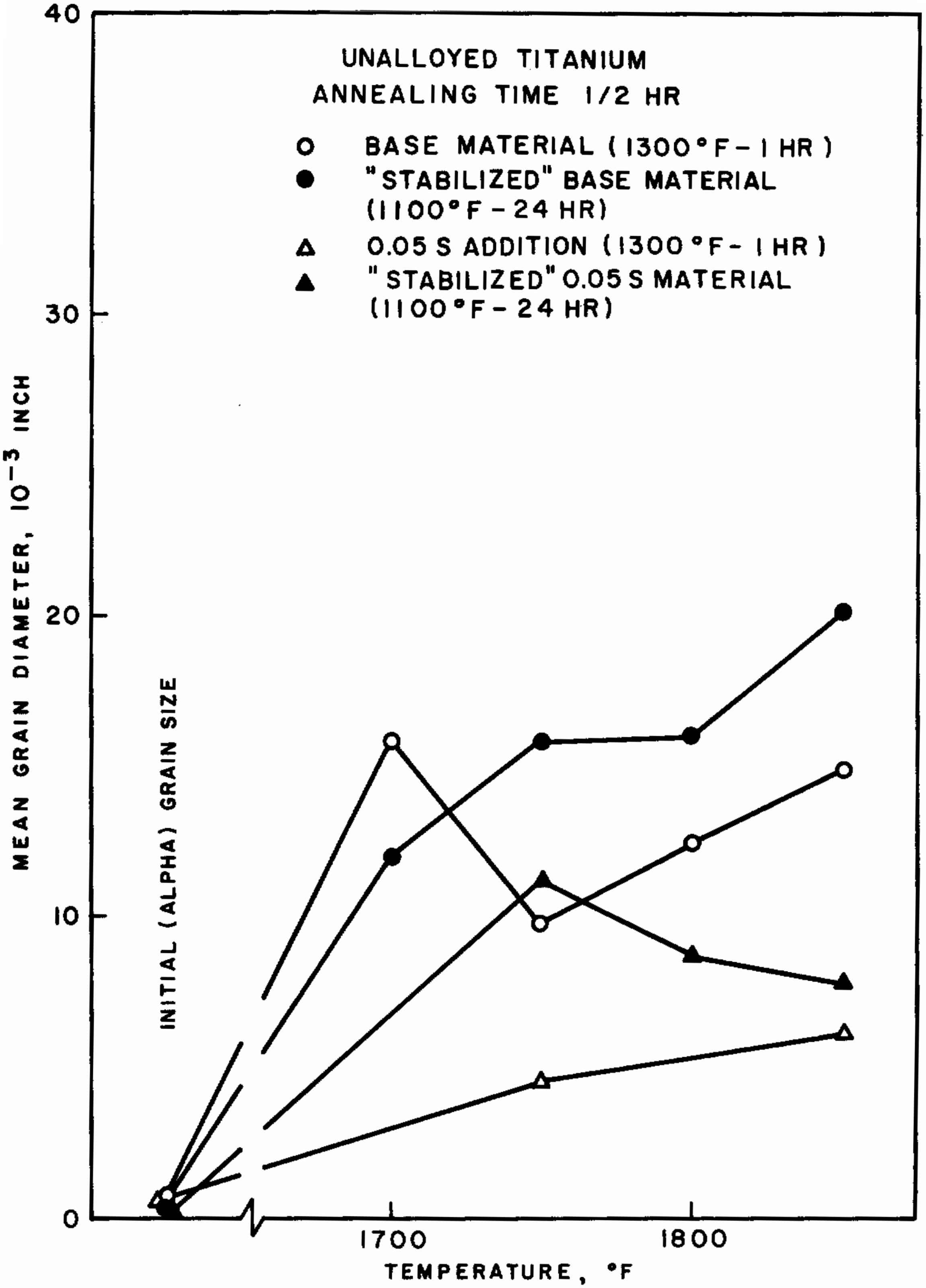


FIG. 19 - COMPARISON OF GRAIN GROWTH OF "STABILIZED" AND STANDARD CONDITIONS OF Ti-BASE AND Ti-0.05S ALLOY FOR 1/2-HOUR ANNEALS.

Approved for Public Release

Contrails

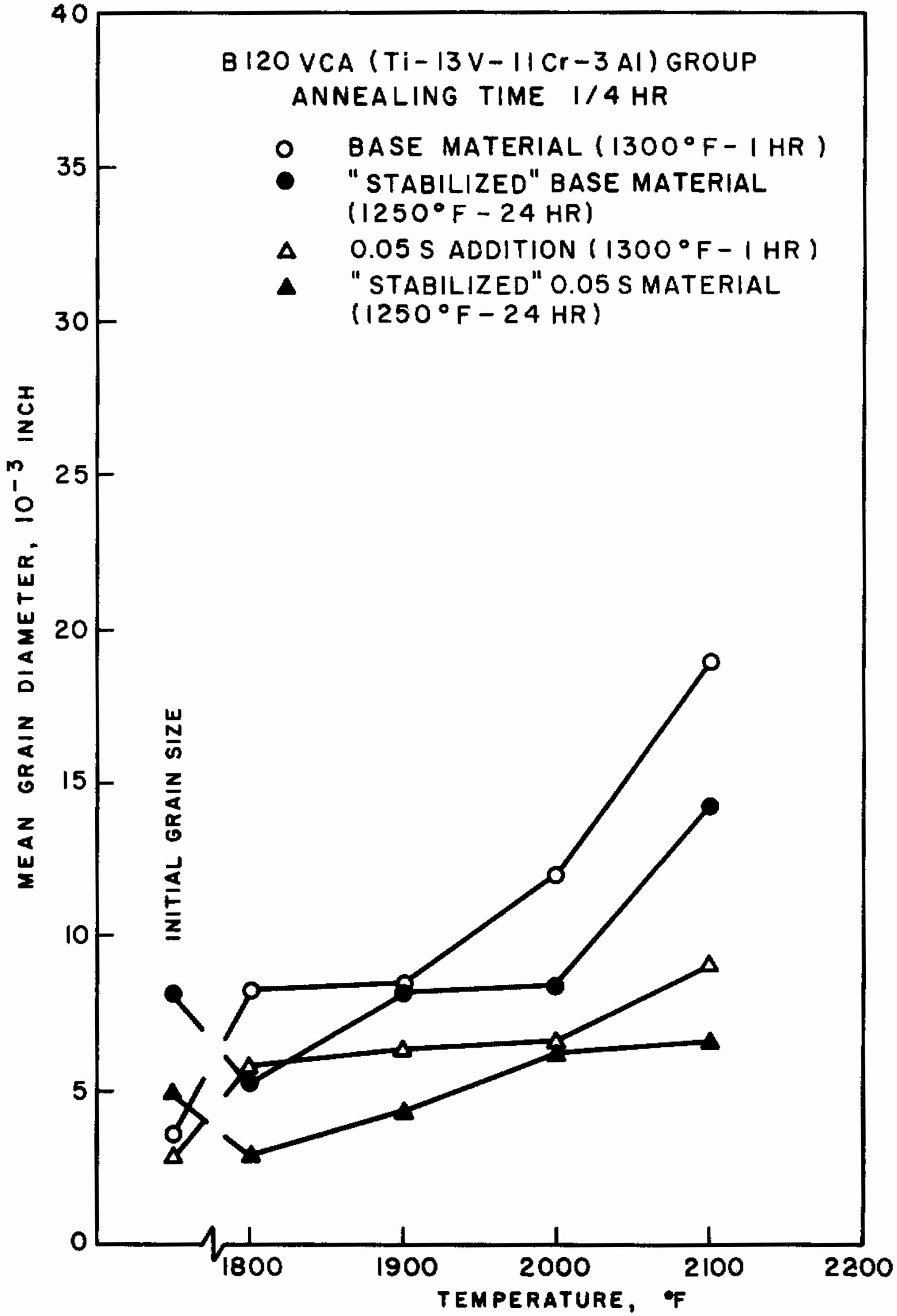


FIG. 20 - COMPARISON OF GRAIN GROWTH OF "STABILIZED" AND STANDARD CONDITIONS OF B-120VCA BASE AND B-120VCA-0.05S ALLOY FOR 1/4-HOUR ANNEALS.

Approved for Public Release

Contrails

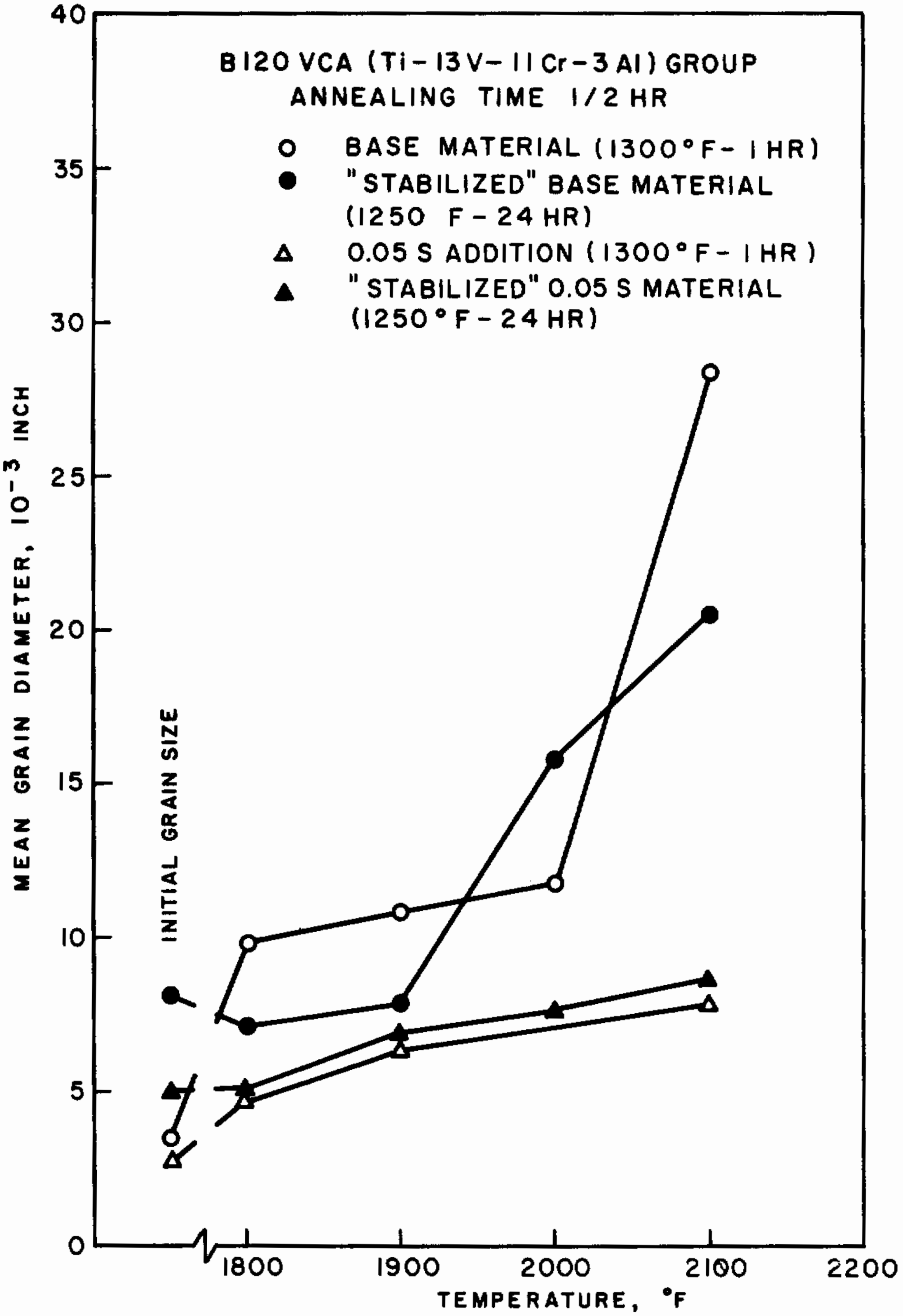
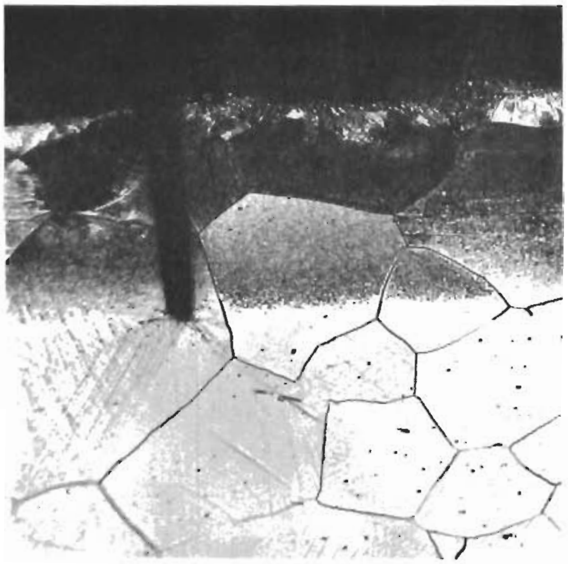


FIG. 21 - COMPARISON OF GRAIN GROWTH OF "STABILIZED" AND STANDARD CONDITIONS OF B-120VCA BASE AND B-120VCA-0.05S ALLOY FOR 1/2-HOUR ANNEALS.

Approved for Public Release

Contrails



Neg. No. 22776 X100

Figure 22

Longitudinal section of B1-1 tensile test specimen showing crack in contaminated surface layers.

Approved for Public Release