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(54) **NICKEL-BASE ALLOY HEAT TREATMENTS,
NICKEL-BASE ALLOYS, AND ARTICLES
INCLUDING NICKEL-BASE ALLOYS**

(75) **Inventor: Erin T. McDevitt, Indian Trail, NC (US)**

(73) **Assignee: ATI Properties, Inc., Albany, OR (US)**

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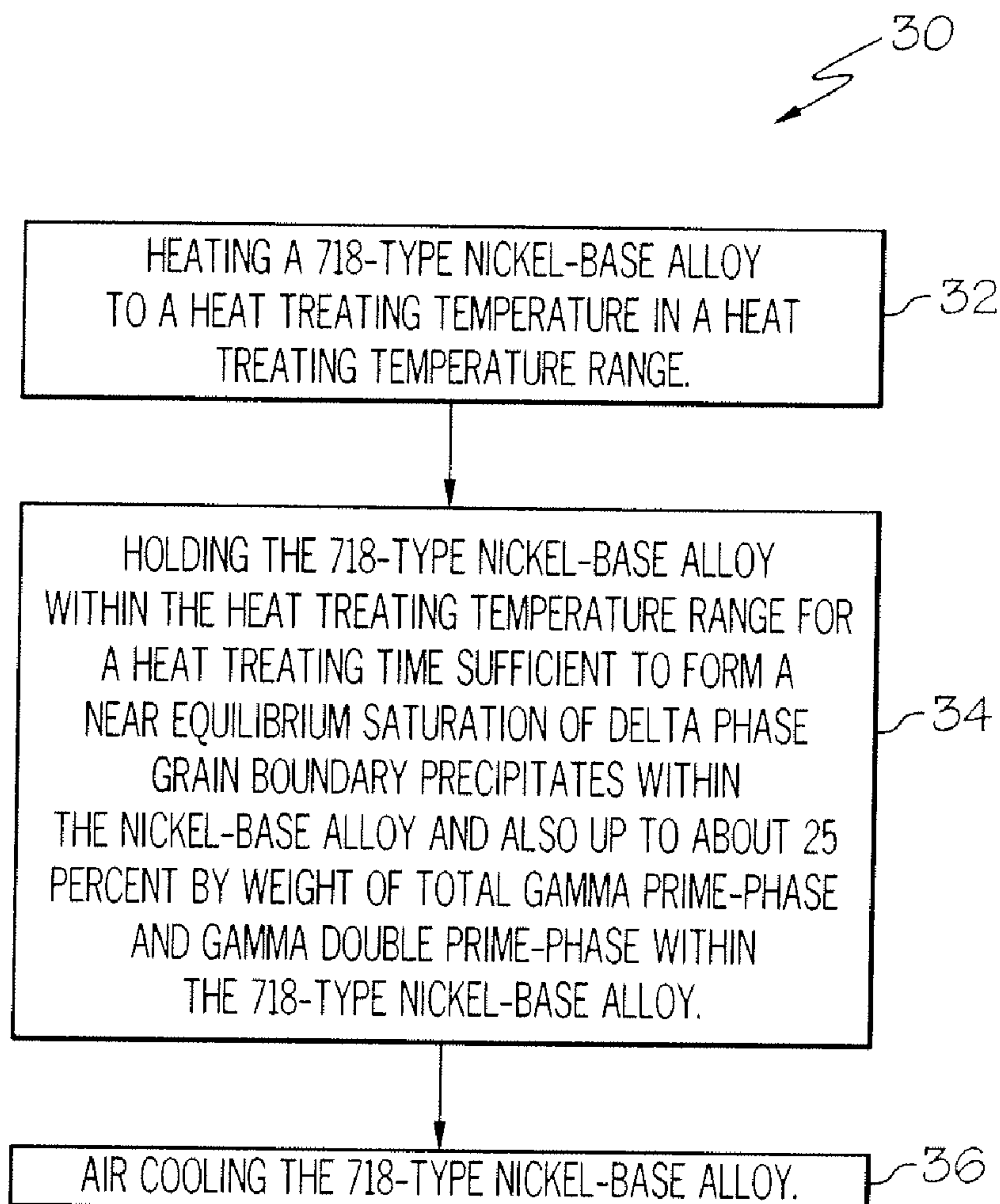
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C22F 1/00 (2006.01)

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USPC **148/707**; 148/675; 148/410; 148/419

(57) **ABSTRACT**

A method for heat treating a 718-type nickel-base comprises heating a 718-type nickel-base alloy to a heat treating temperature, and holding the alloy at the heat treating temperature for a heat treating time sufficient to form an equilibrium or near-equilibrium concentration of δ -phase grain boundary precipitates within the nickel-base alloy and up to 25 percent by weight of total γ' -phase and γ'' -phase. The 718-type nickel-base alloy is air cooled. The present disclosure also includes a 718-type nickel-base alloy comprising a near-equilibrium concentration of δ -phase grain boundary precipitates and up to 25 percent by weight of total γ' -phase and γ'' -phase precipitates. Alloys according to the disclosure may be included in articles of manufacture such as, for example, face sheet, honeycomb core elements, and honeycomb panels for thermal protection systems for hypersonic flight vehicles and space vehicles.



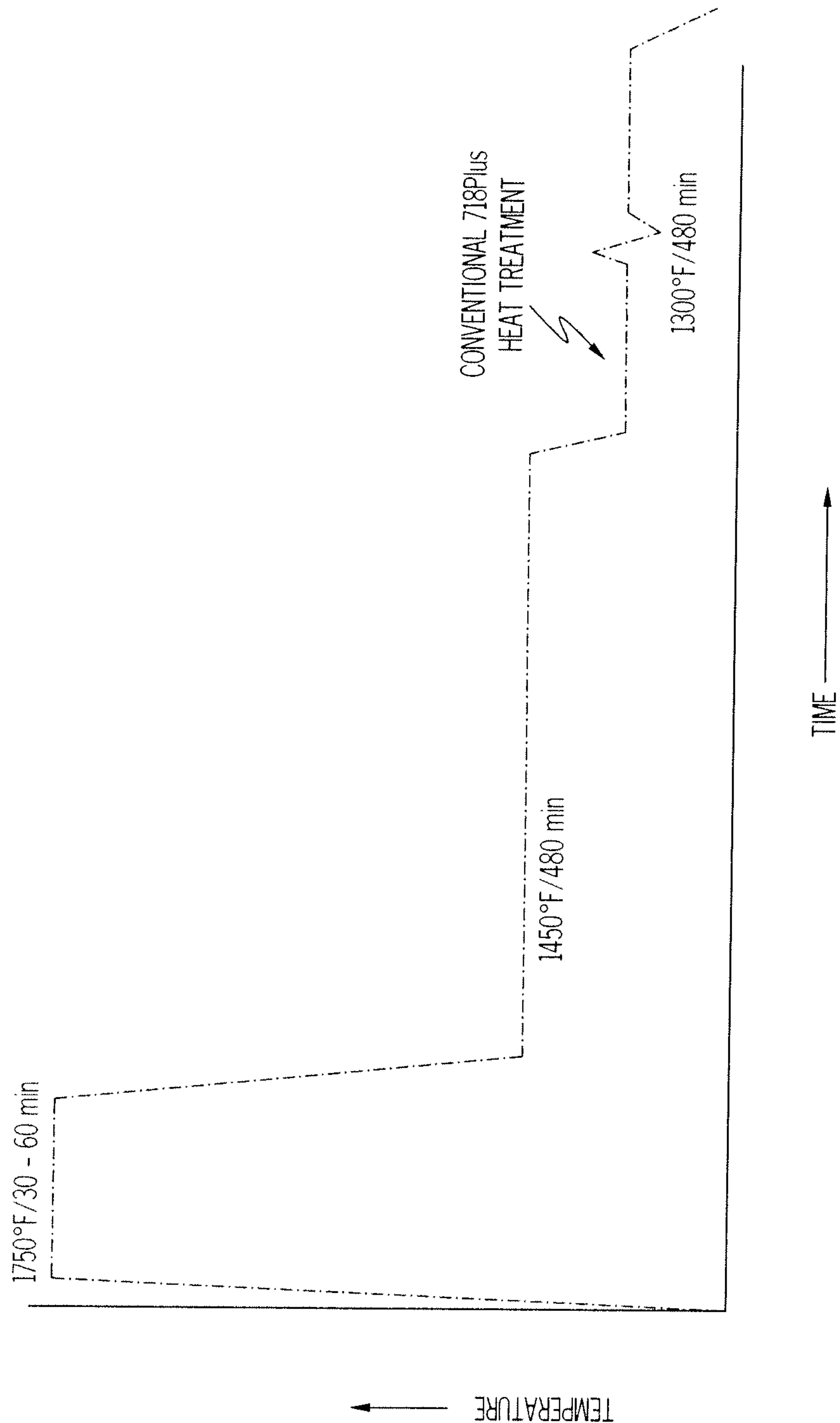


FIG. 1
(PRIOR ART)

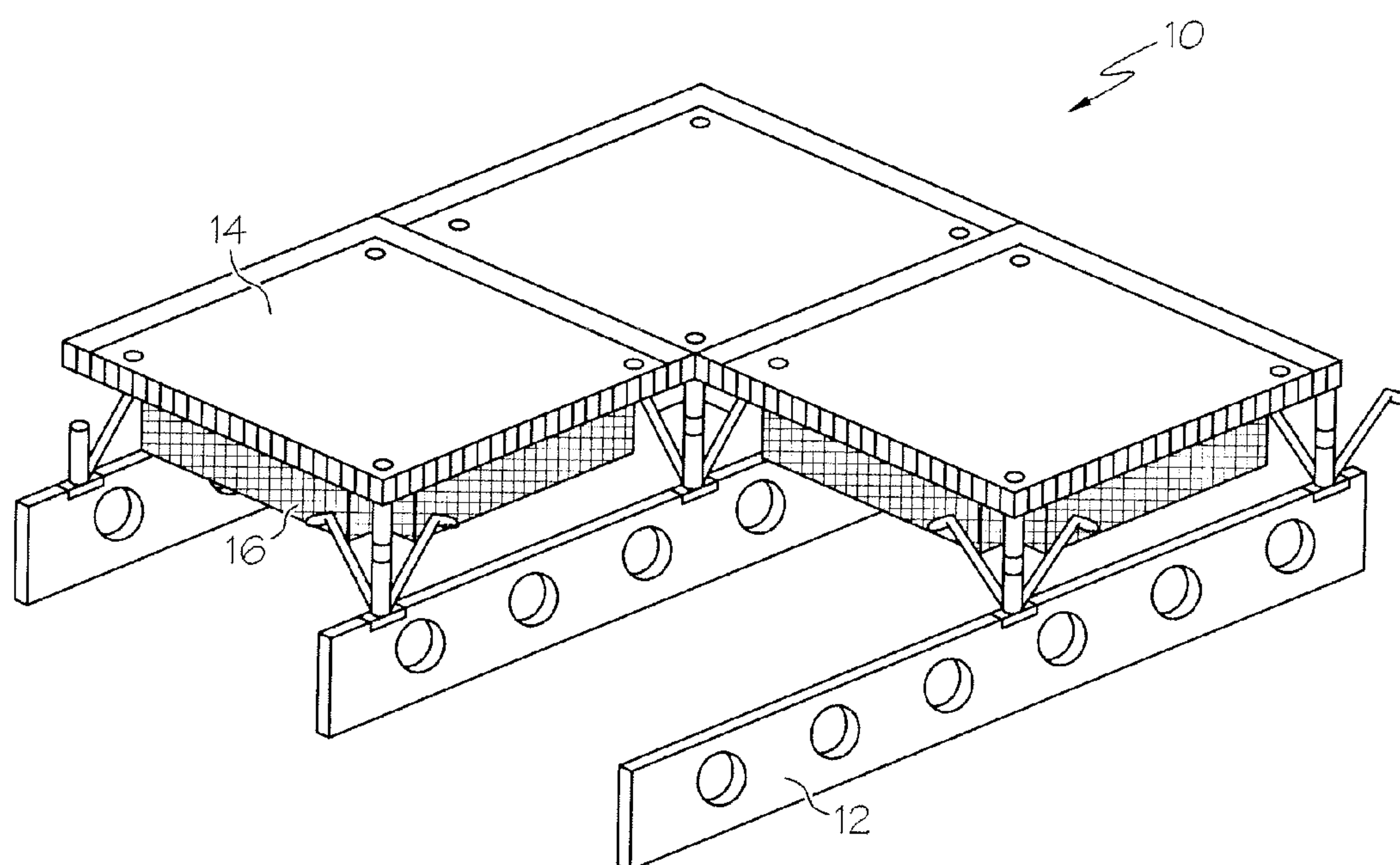


FIG. 2
(PRIOR ART)

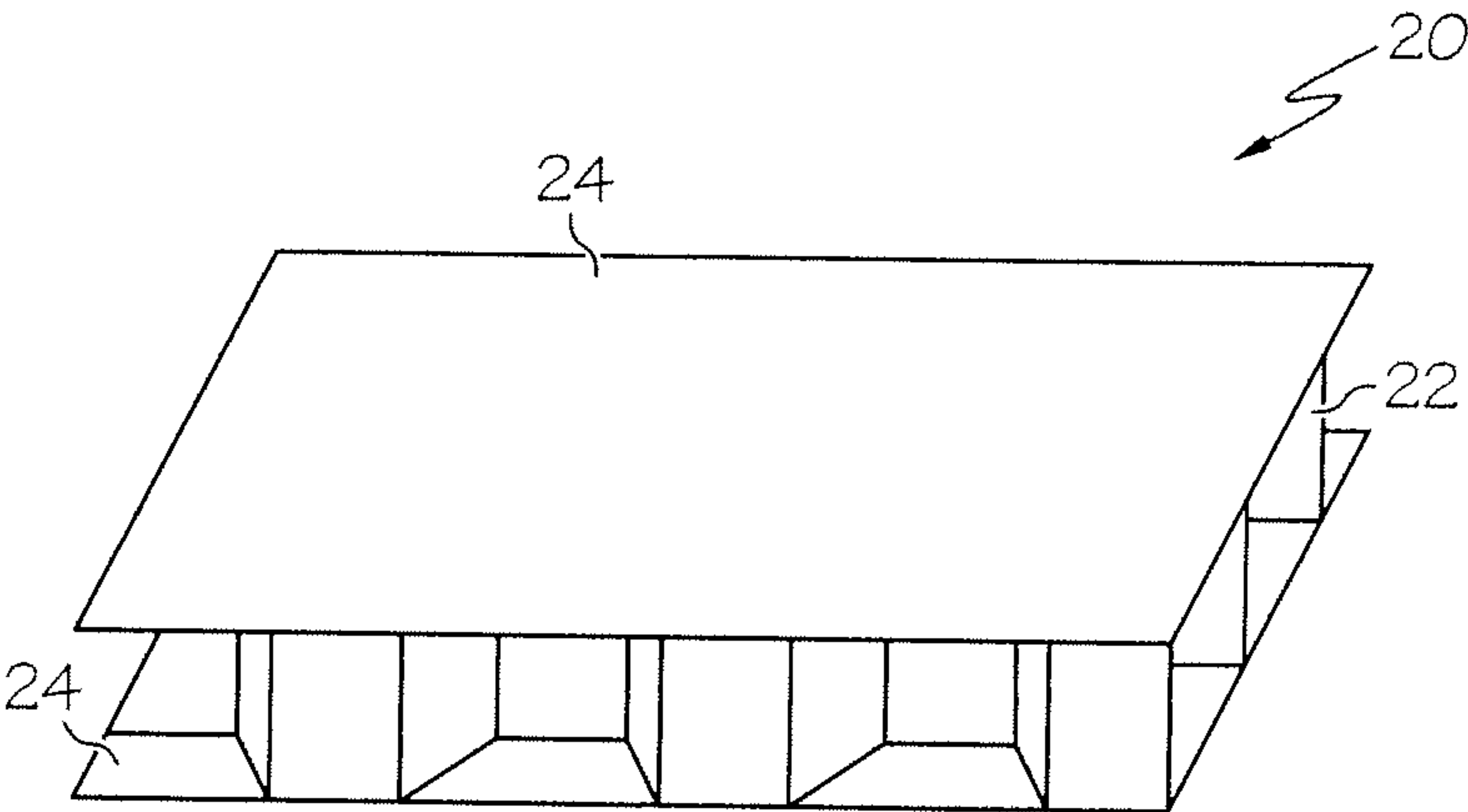


FIG. 3A
(PRIOR ART)

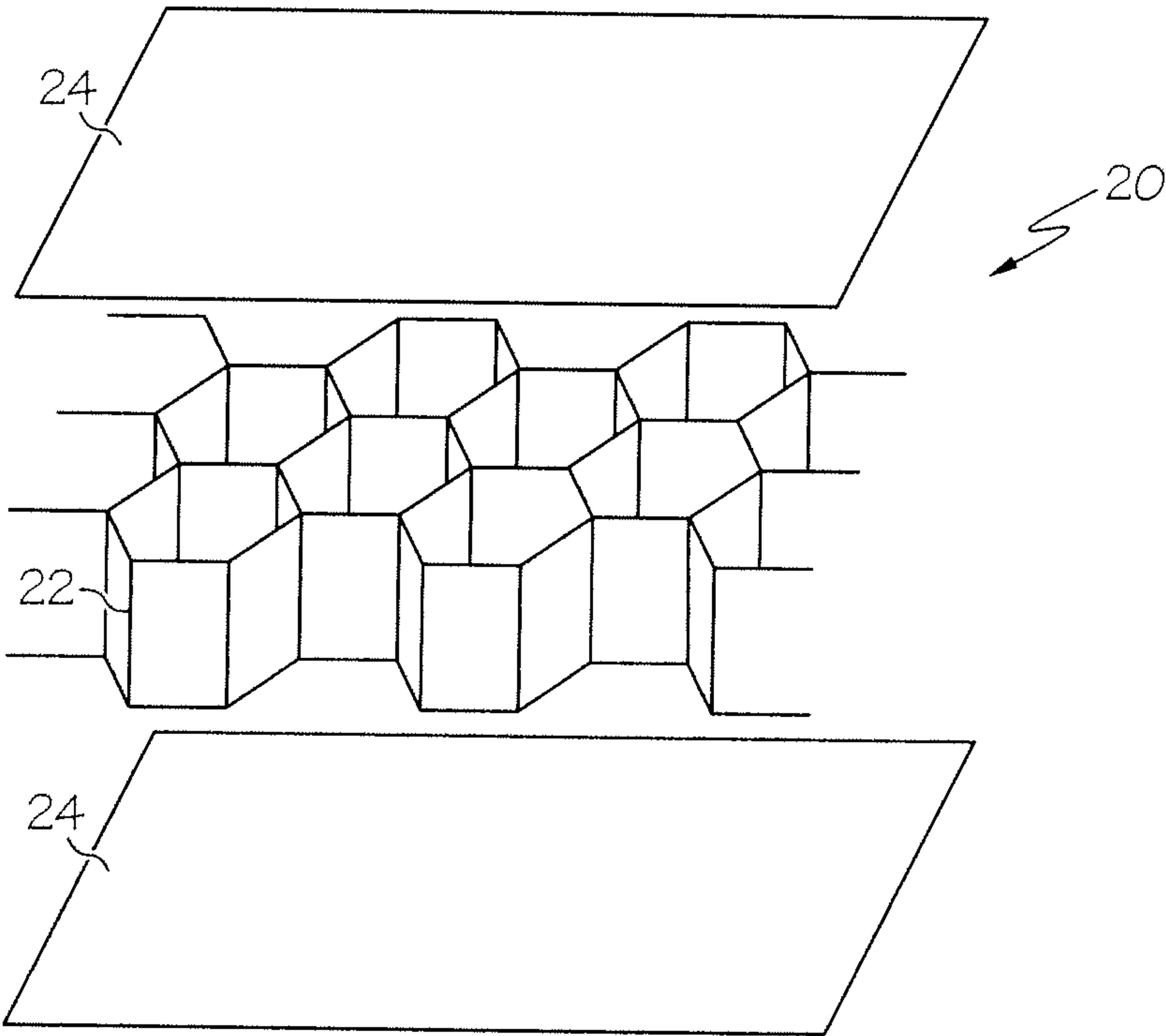


FIG. 3B
(PRIOR ART)

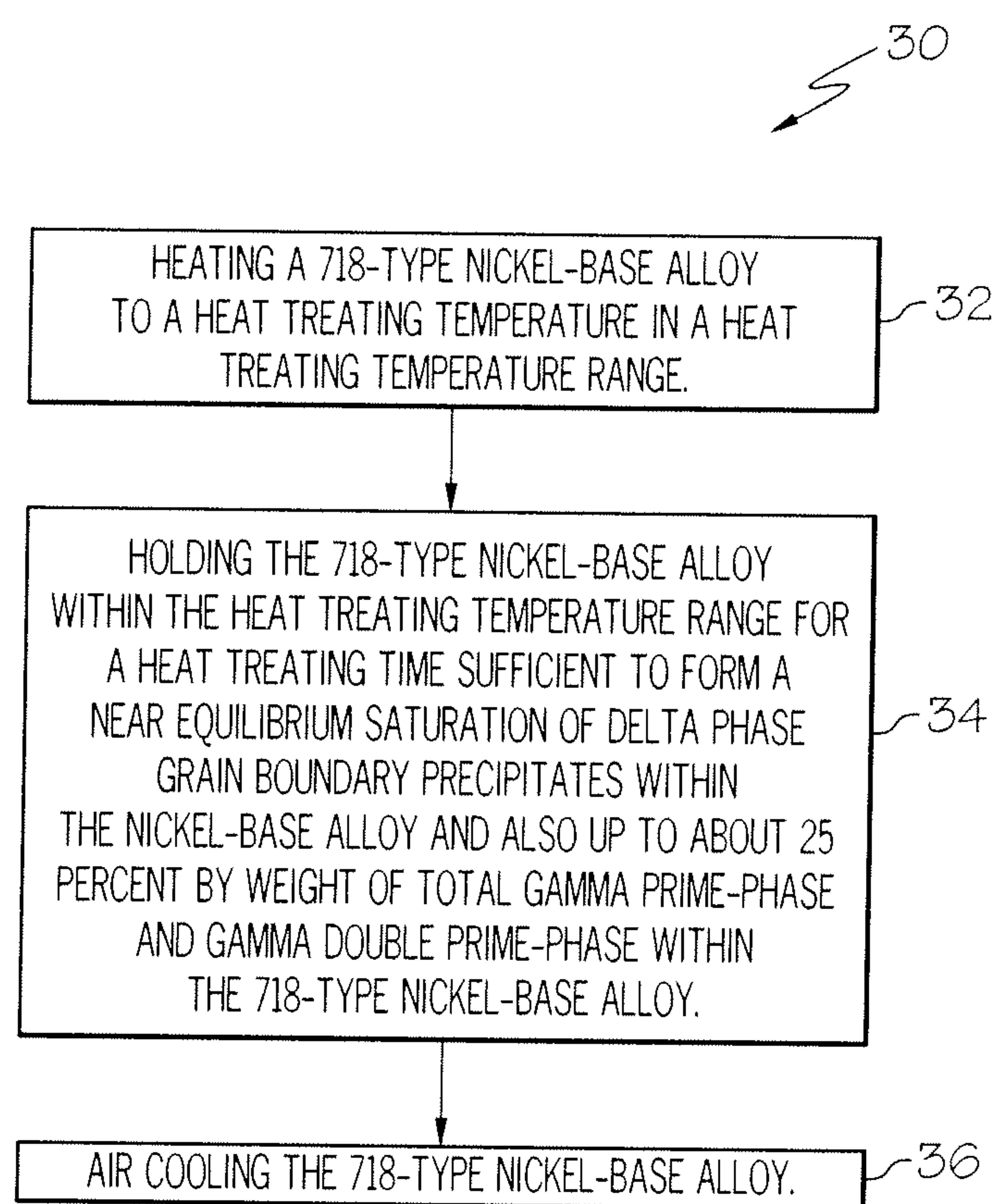


FIG. 4

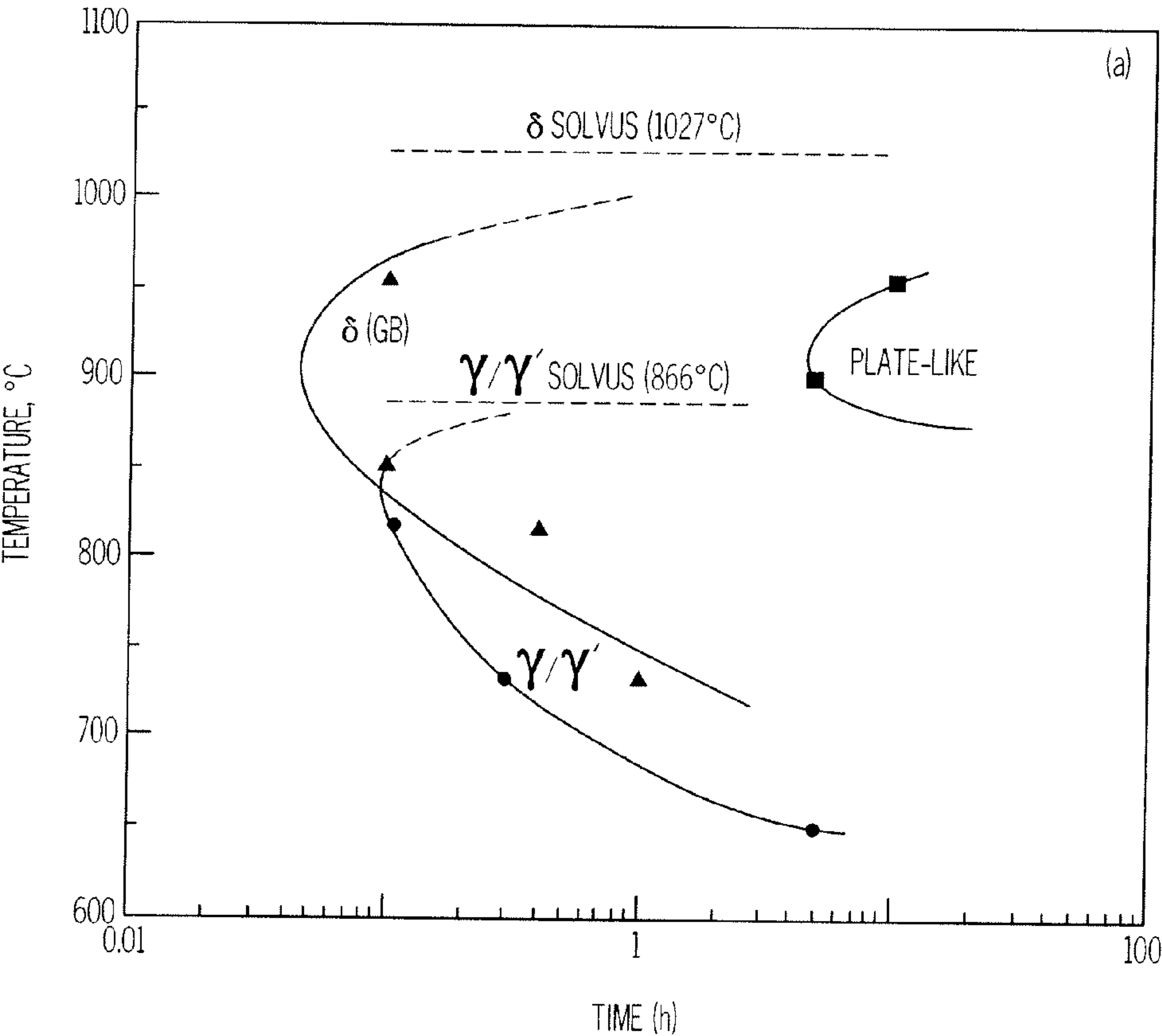


FIG. 5A

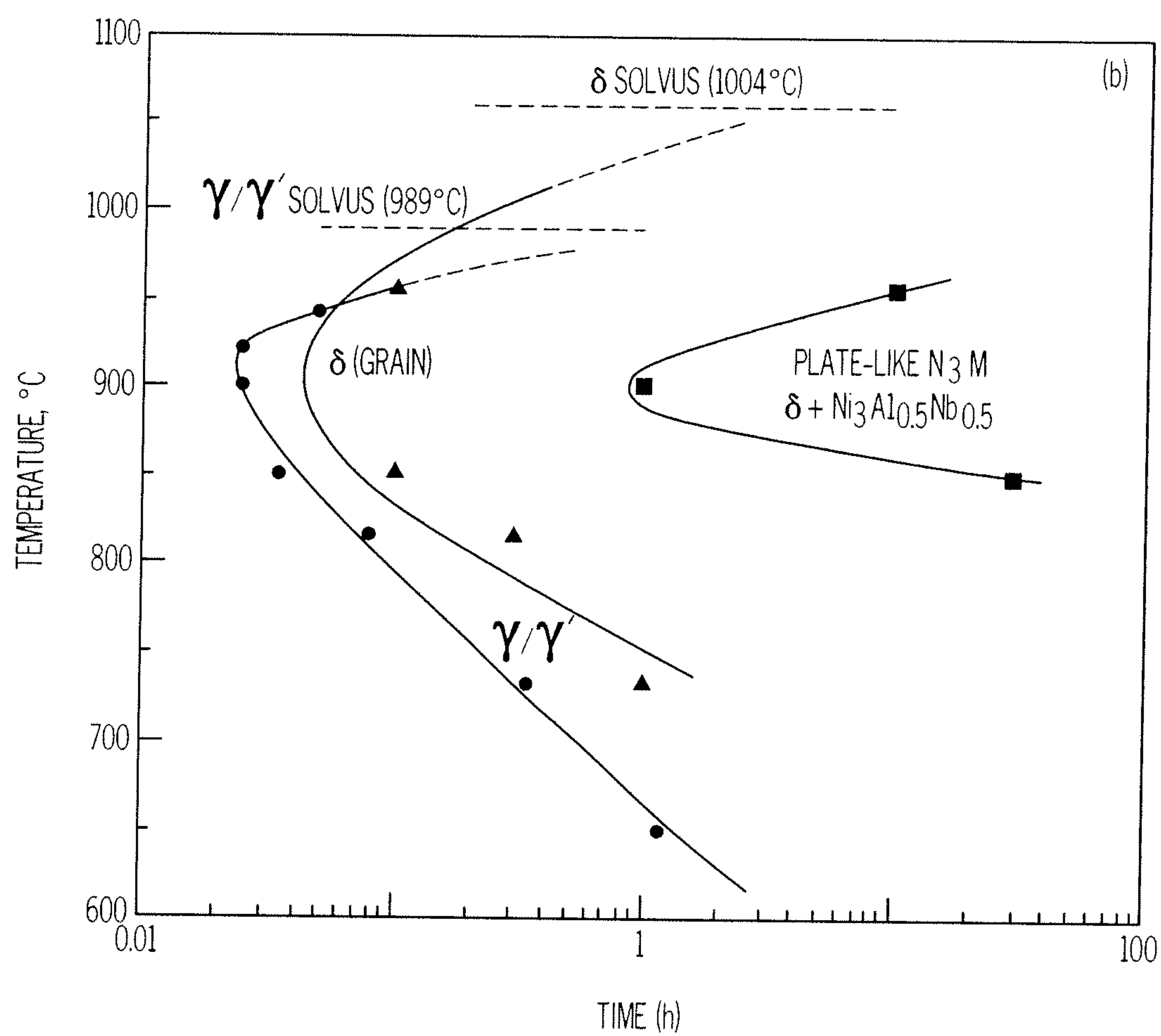


FIG. 5B

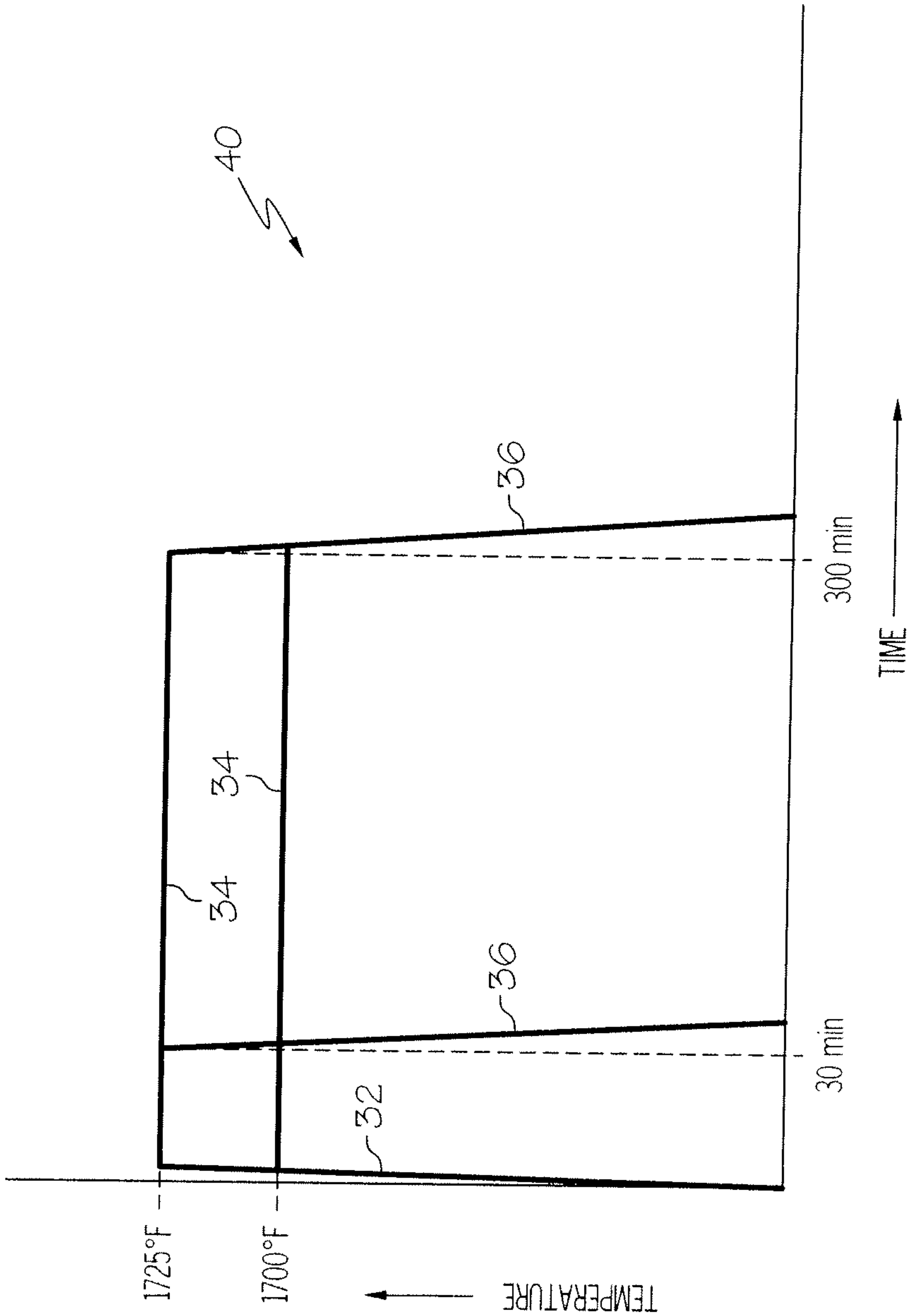


FIG. 6

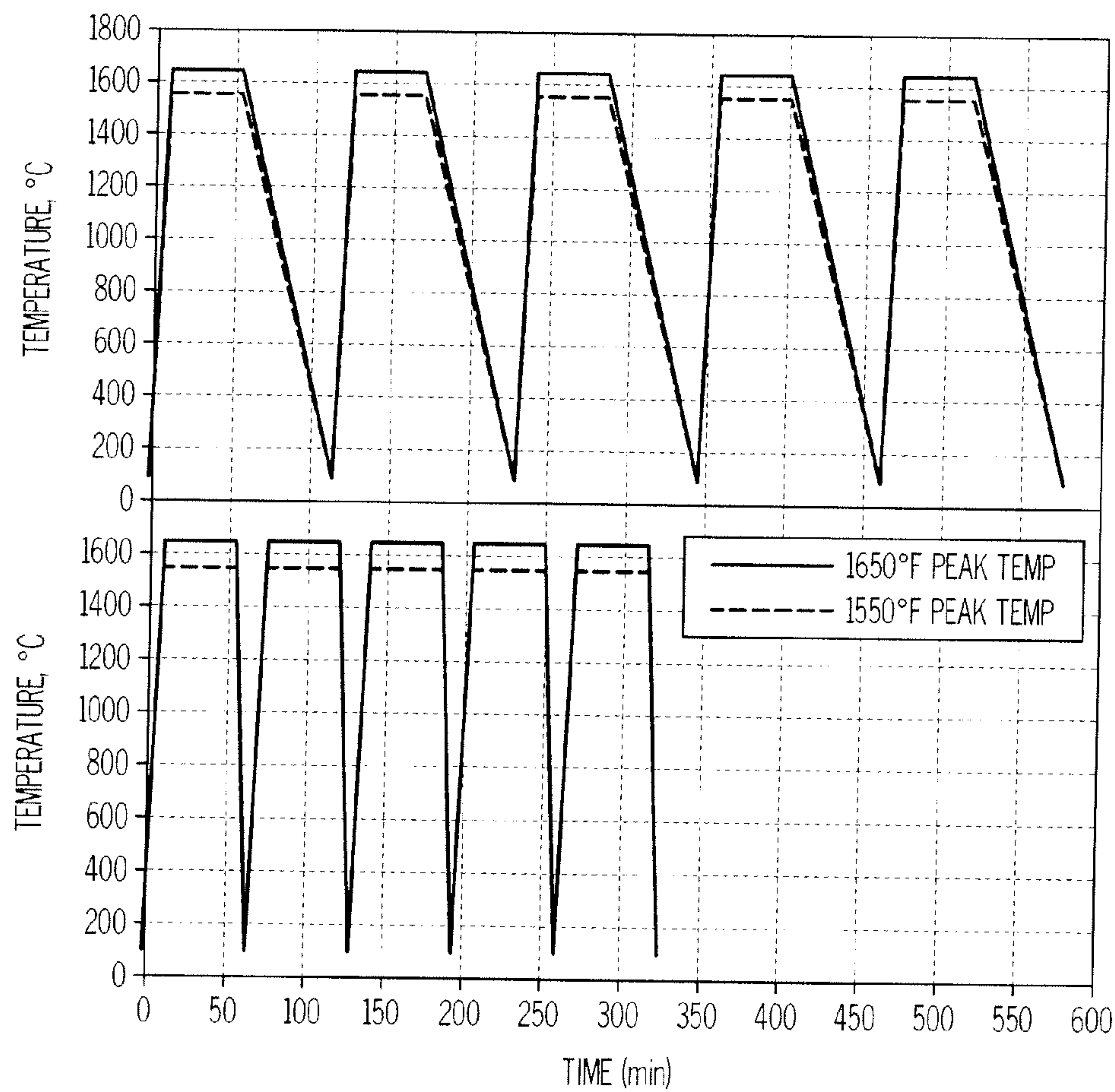


FIG. 7

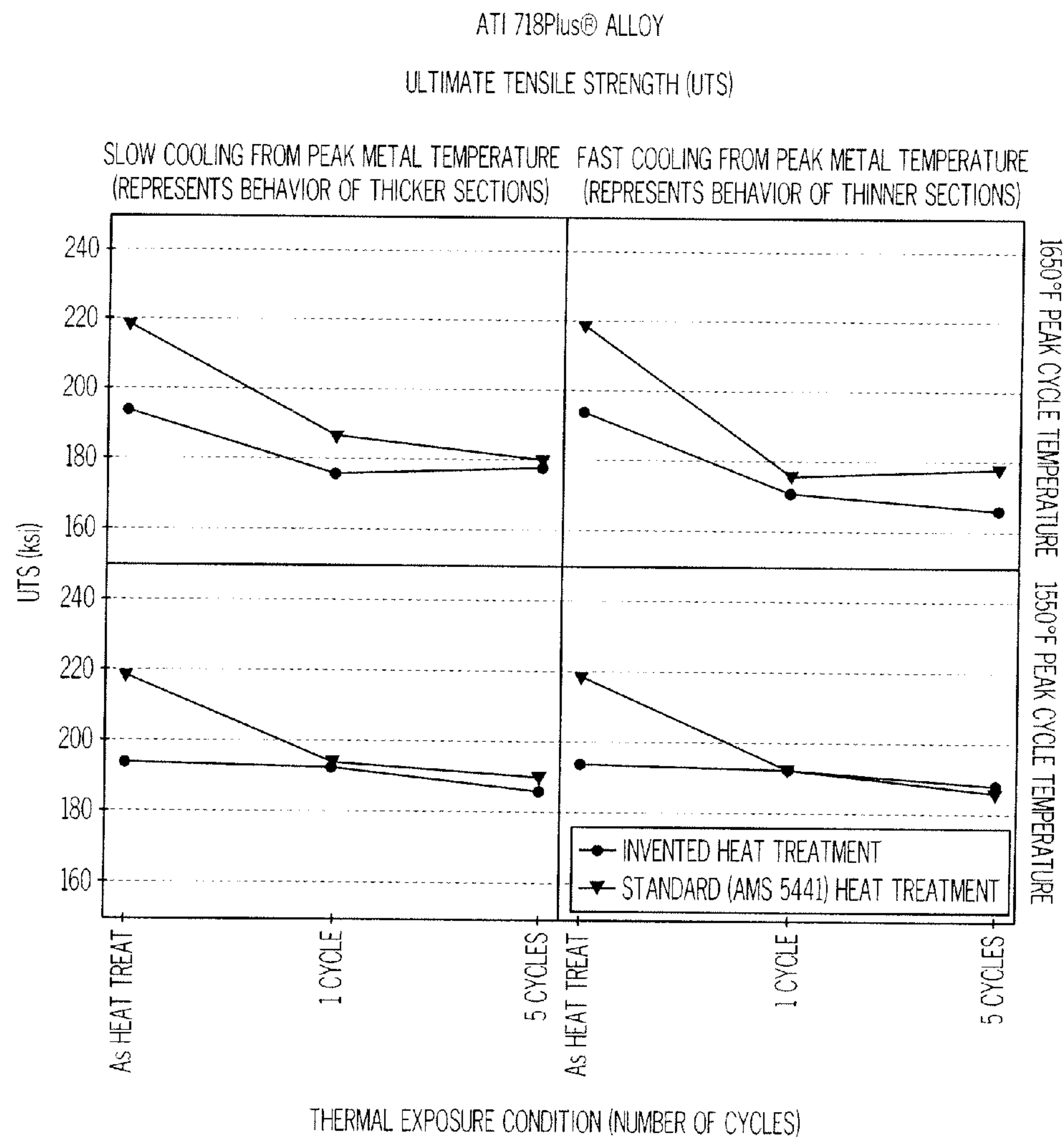


FIG. 8

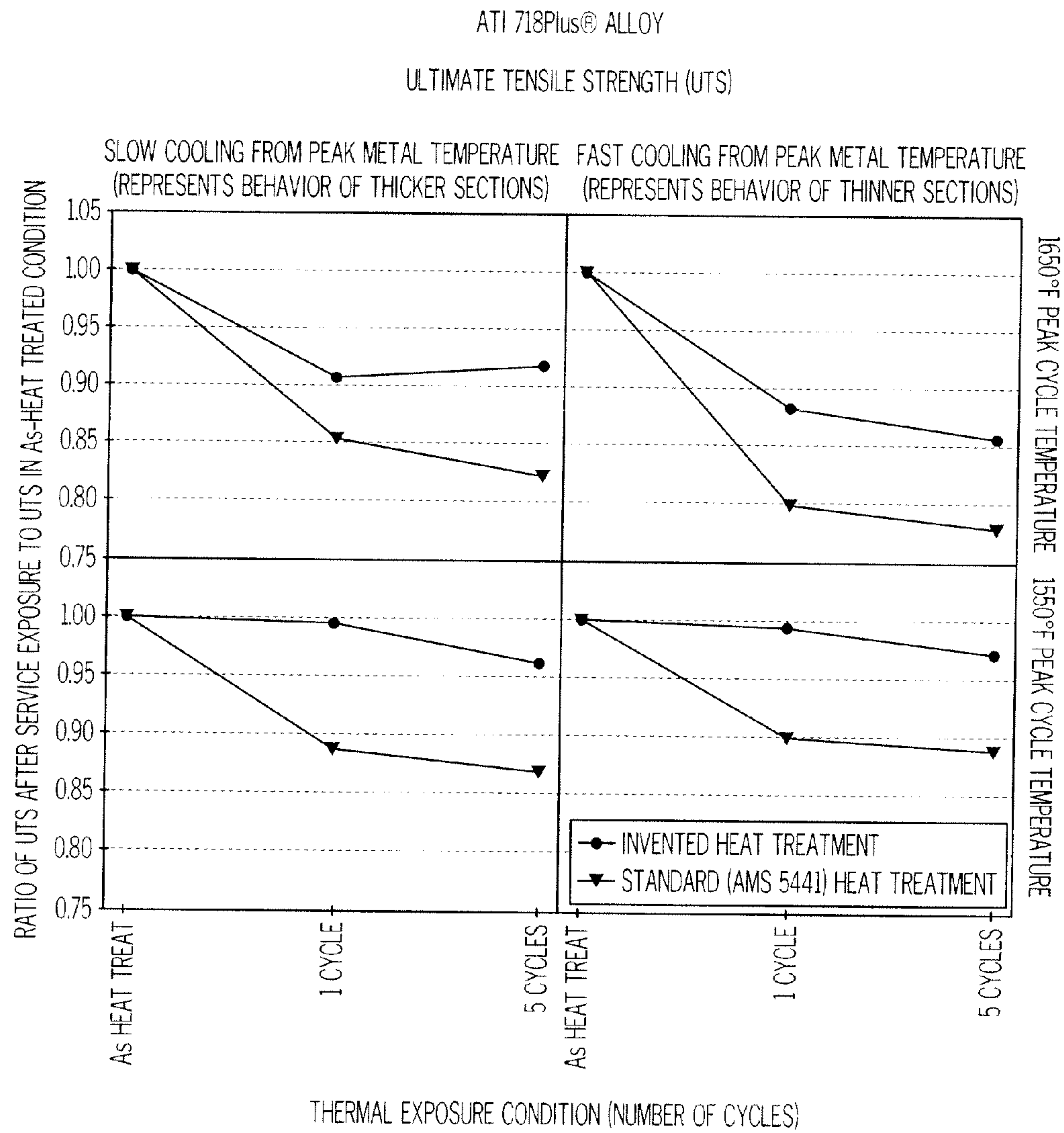


FIG. 9

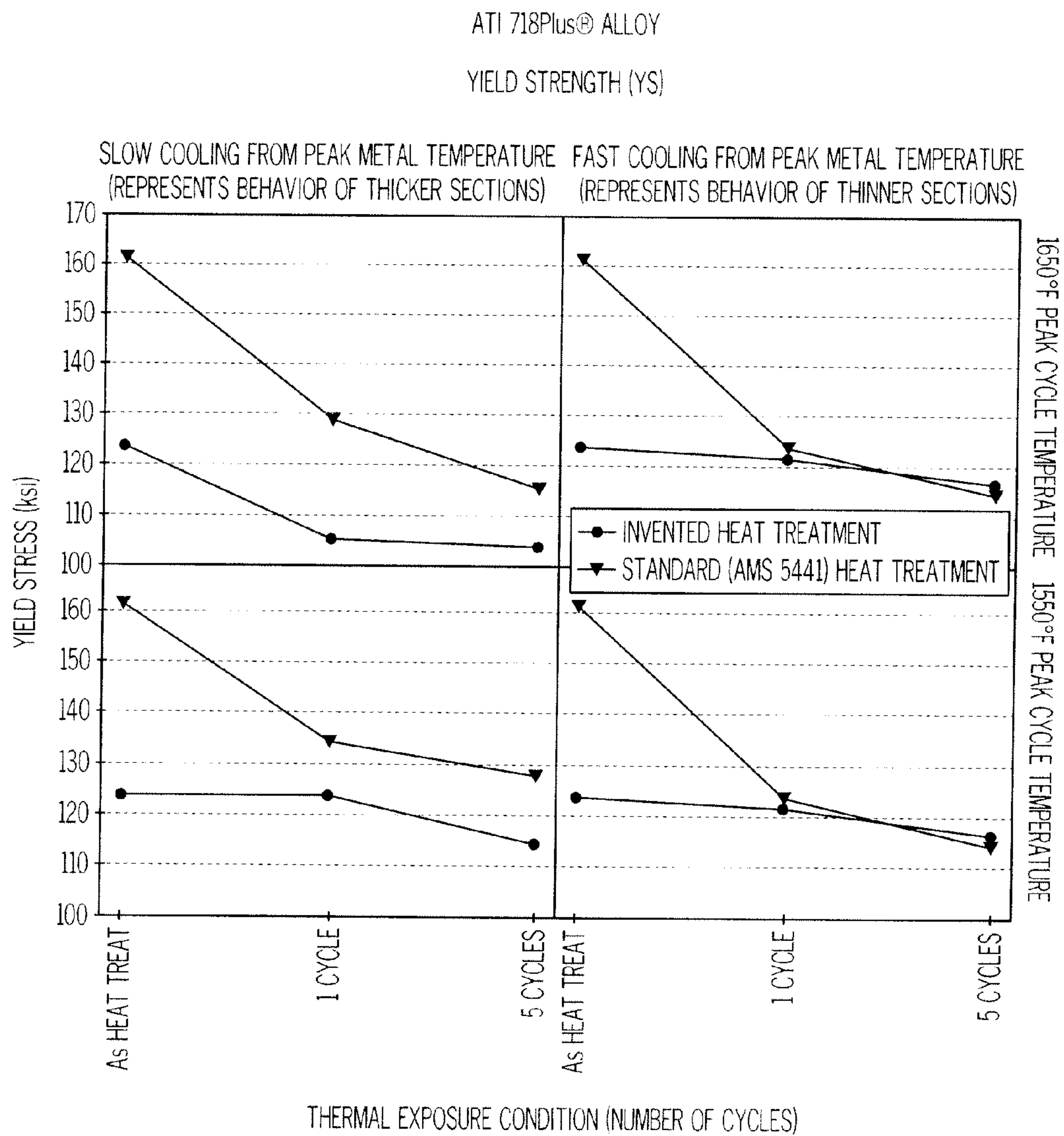


FIG. 10

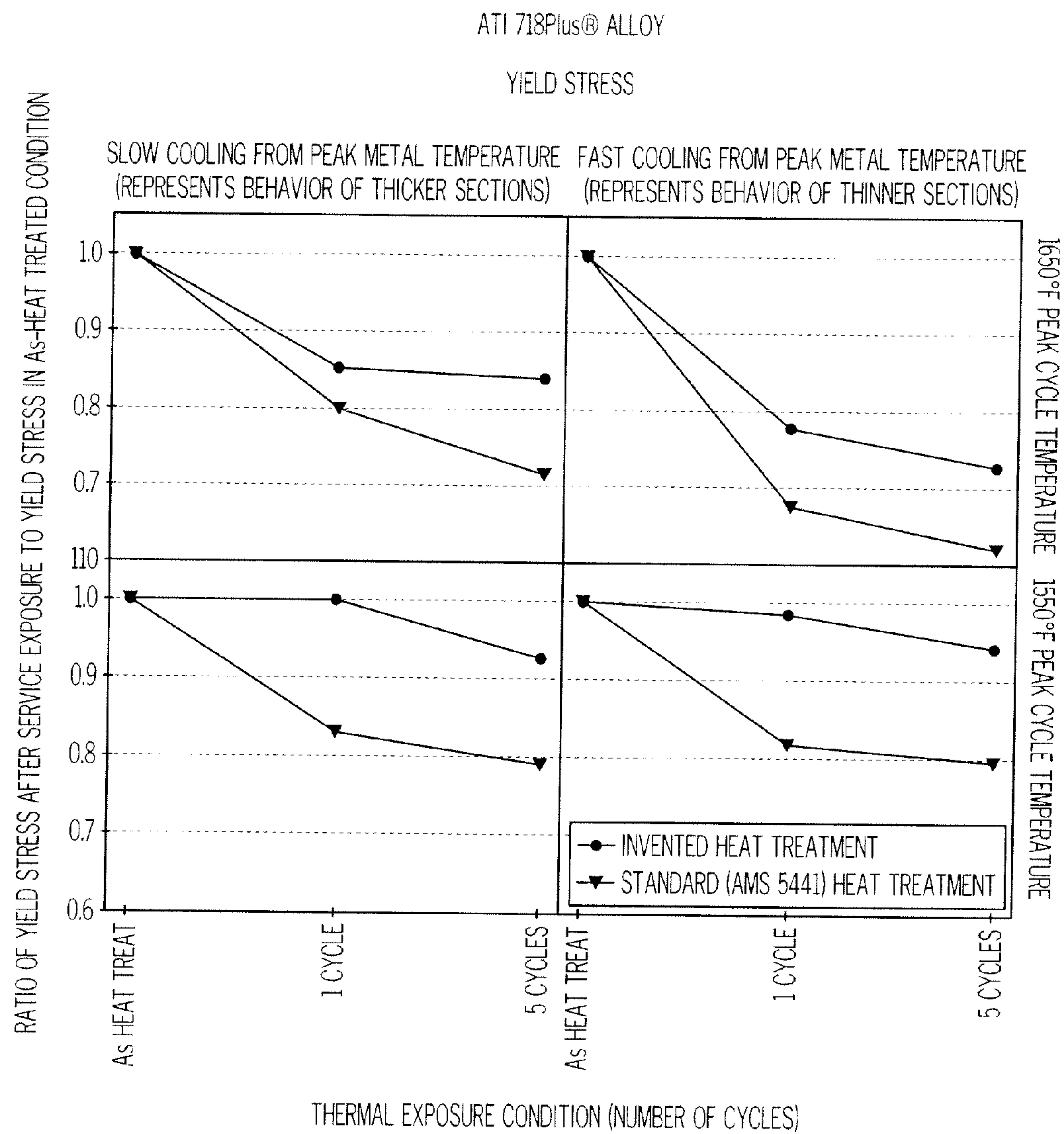


FIG. 11

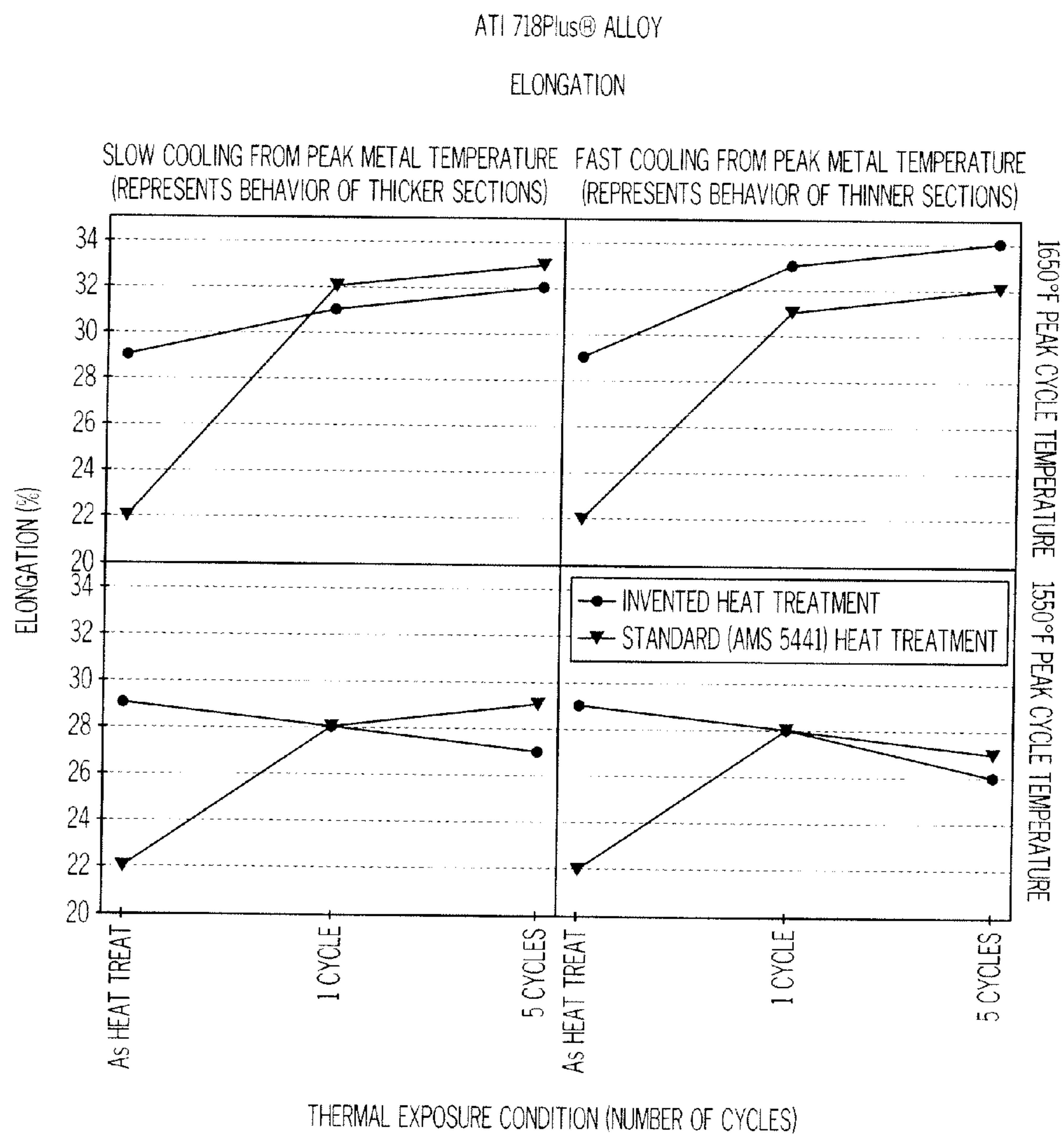


FIG. 12

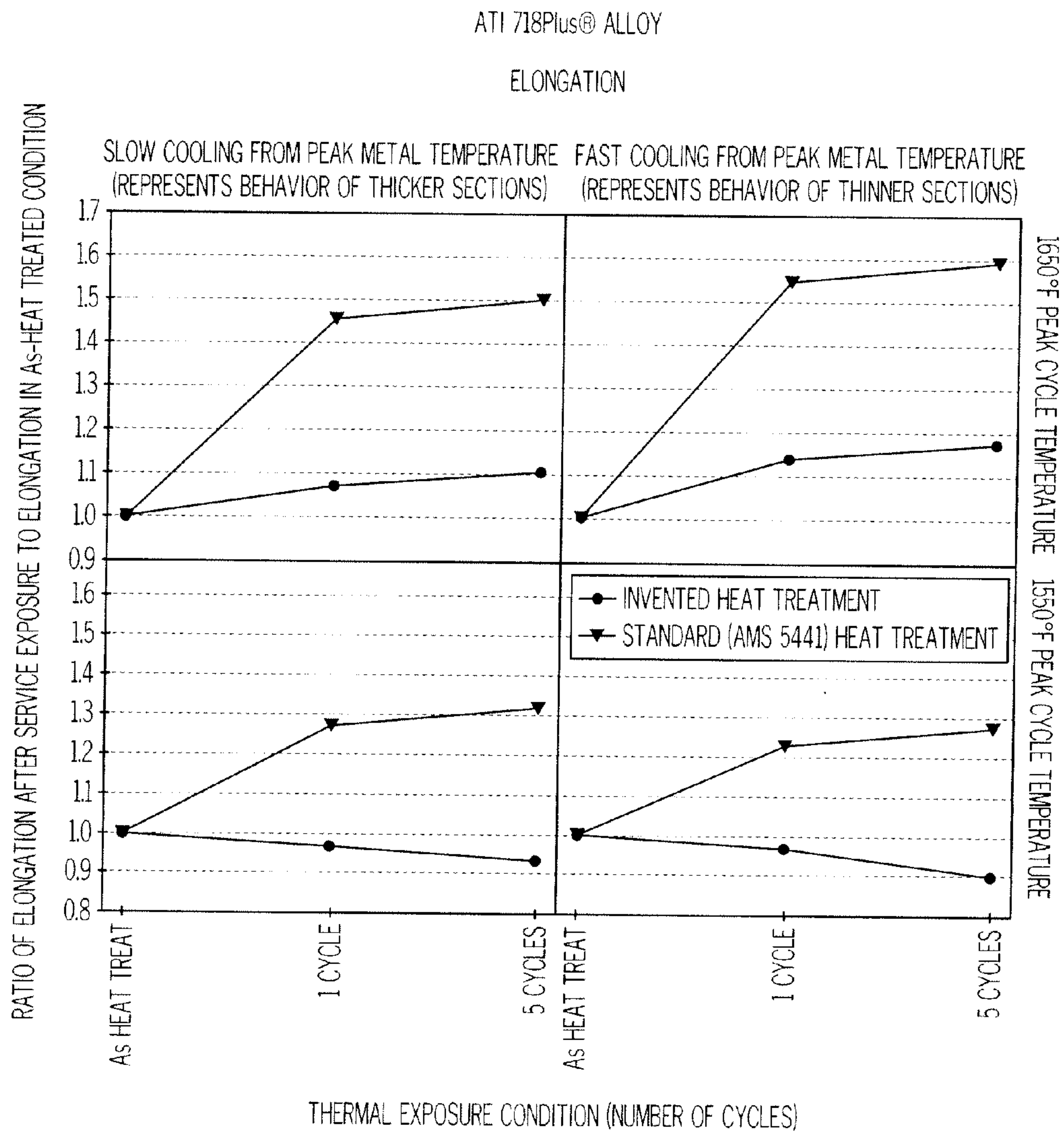


FIG. 13

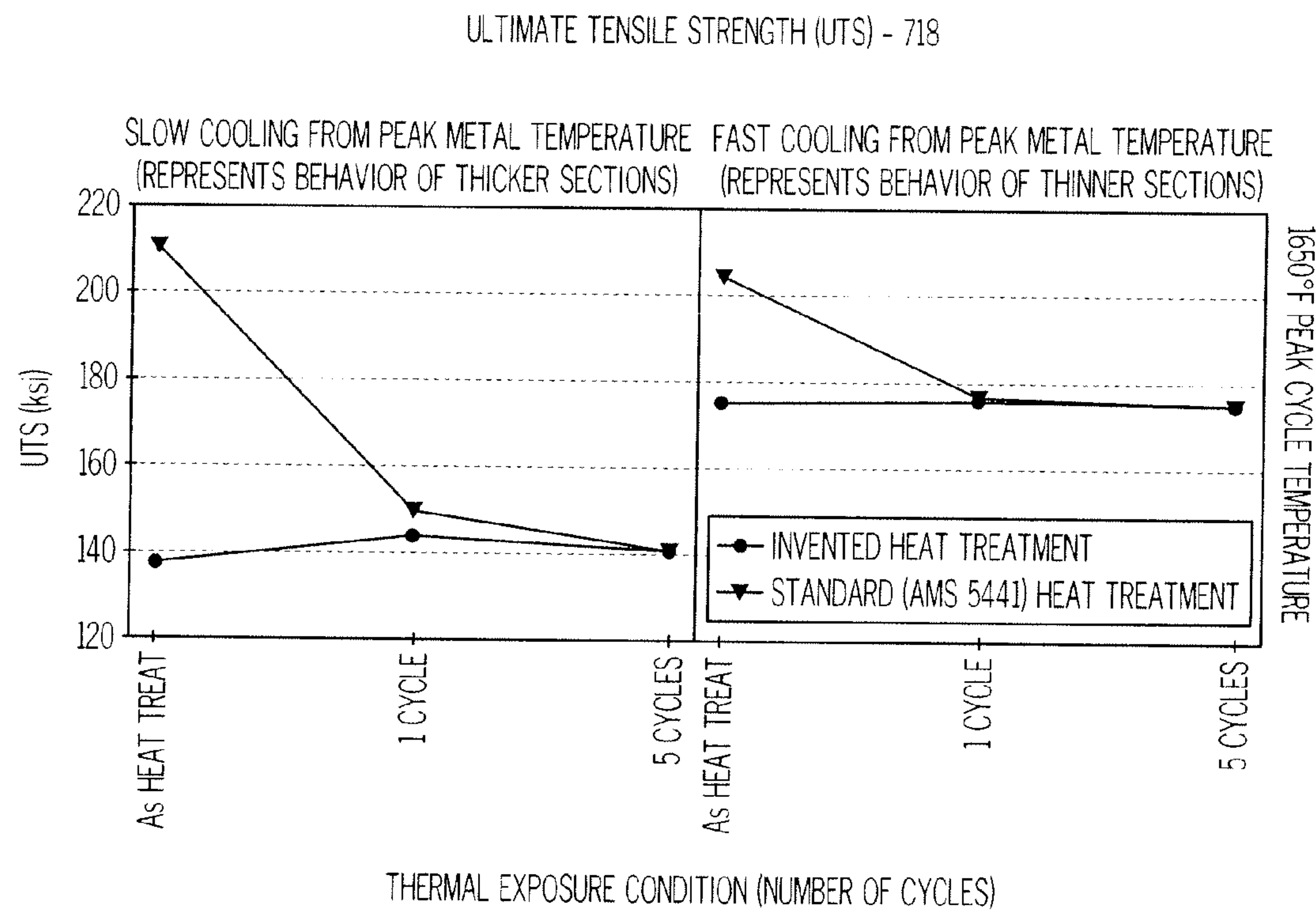


FIG. 14

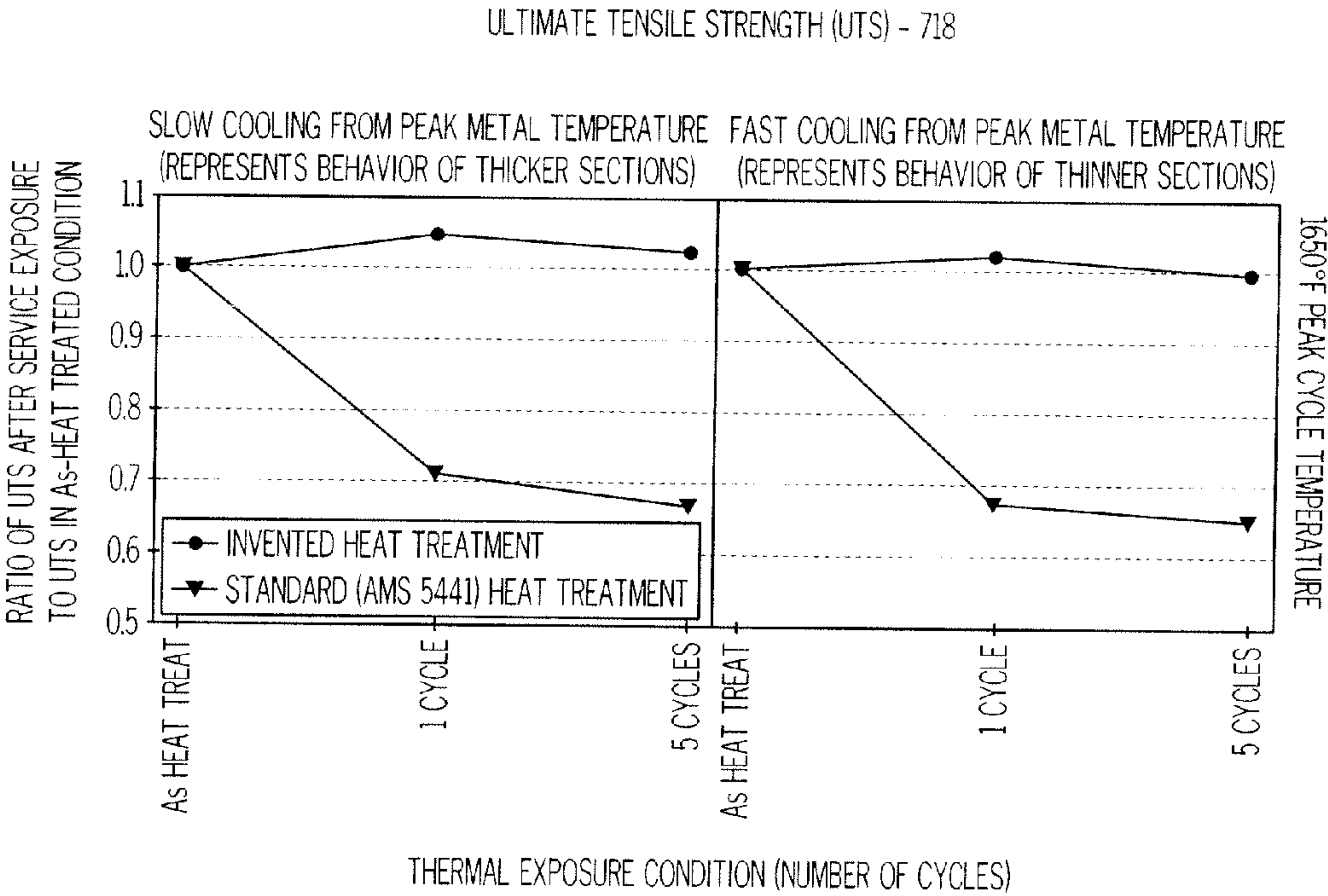


FIG. 15

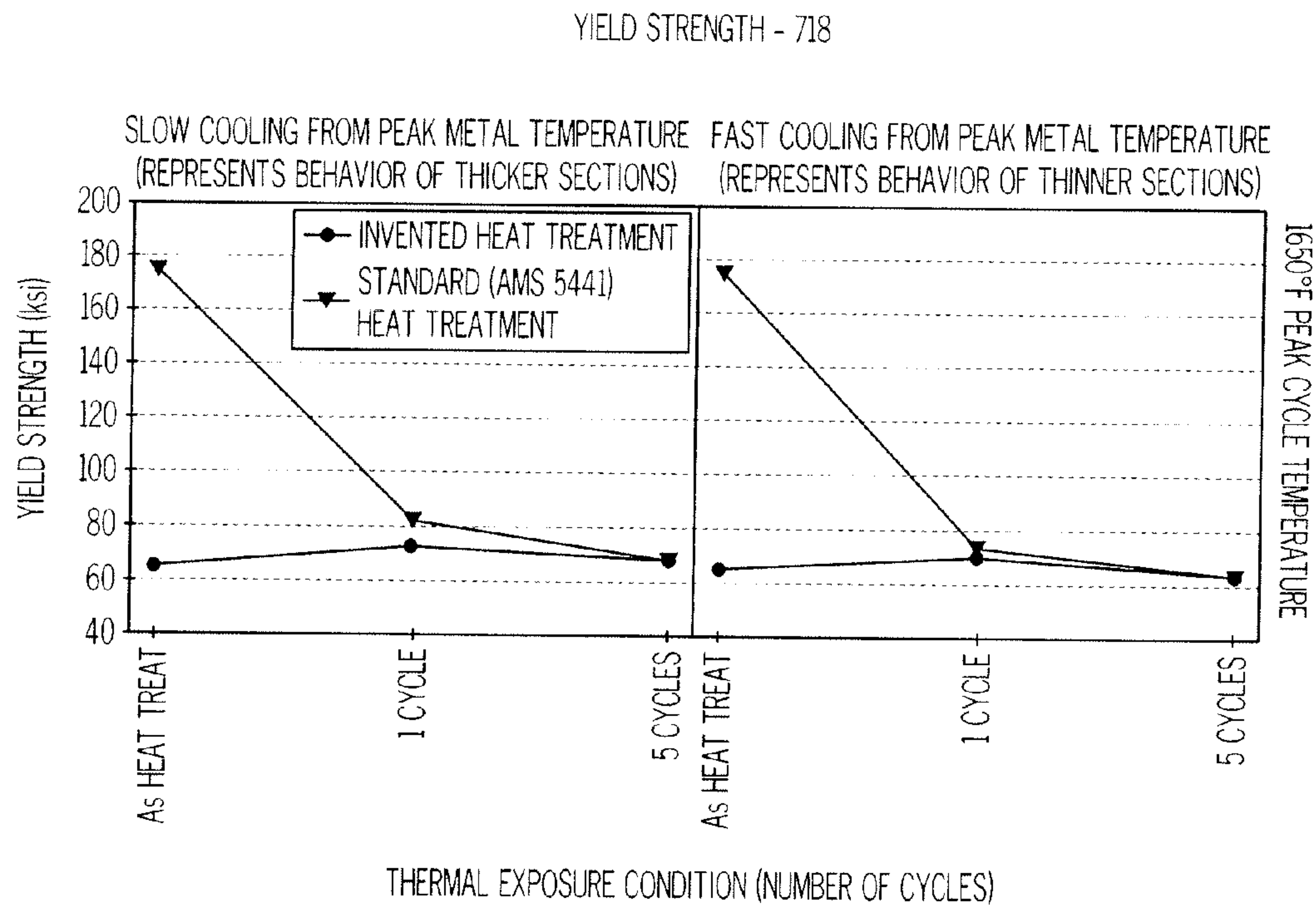


FIG. 16

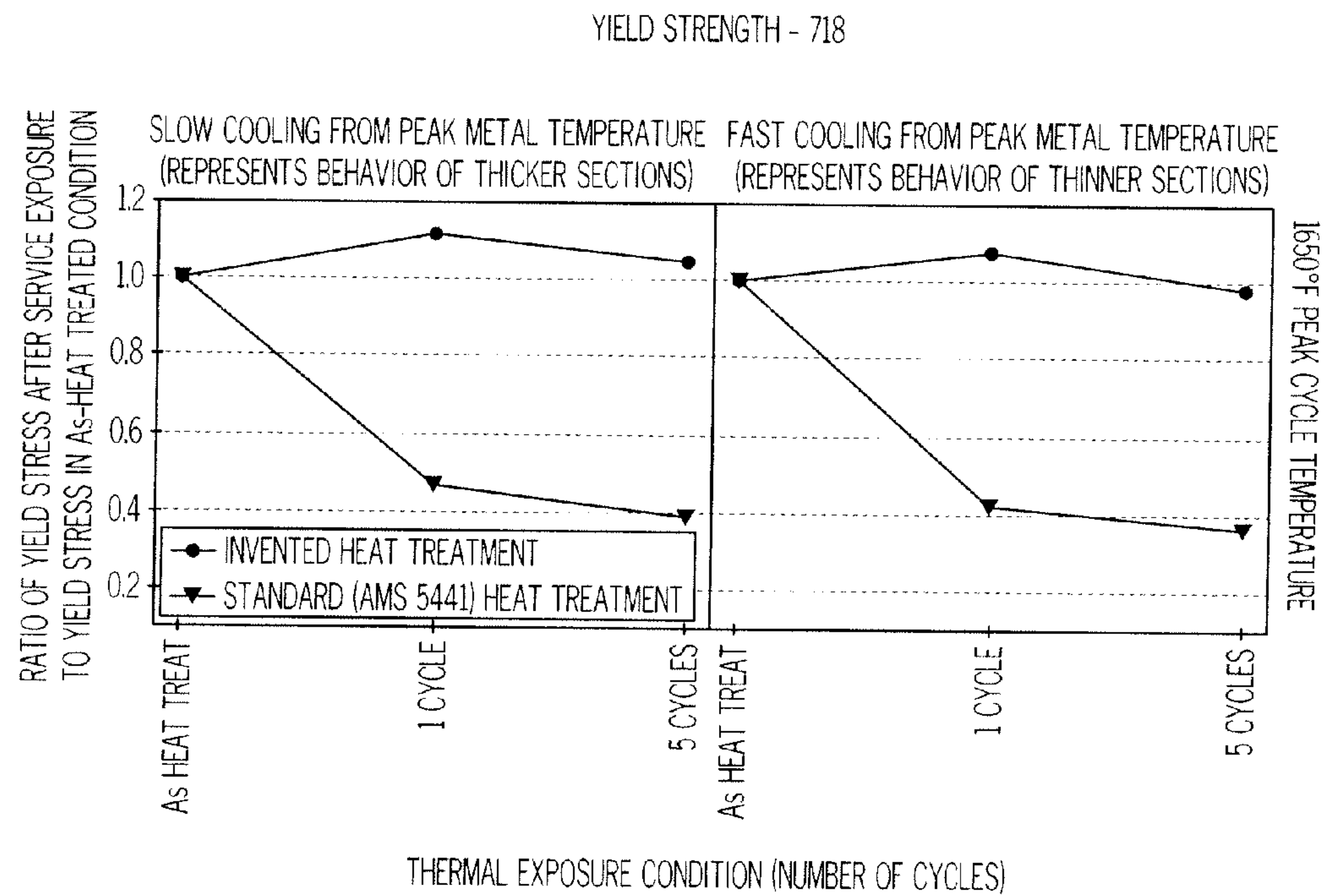


FIG. 17

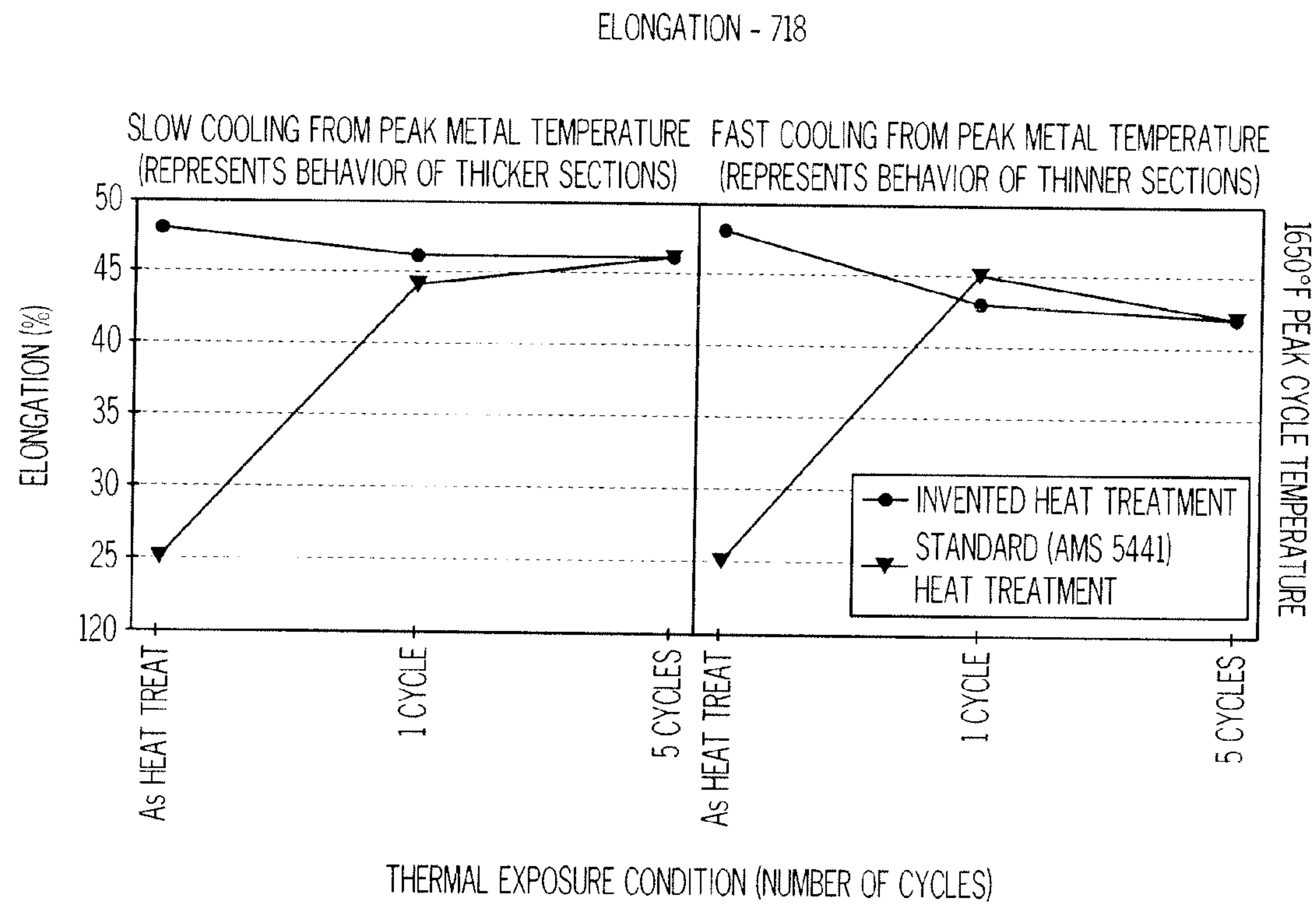


FIG. 18

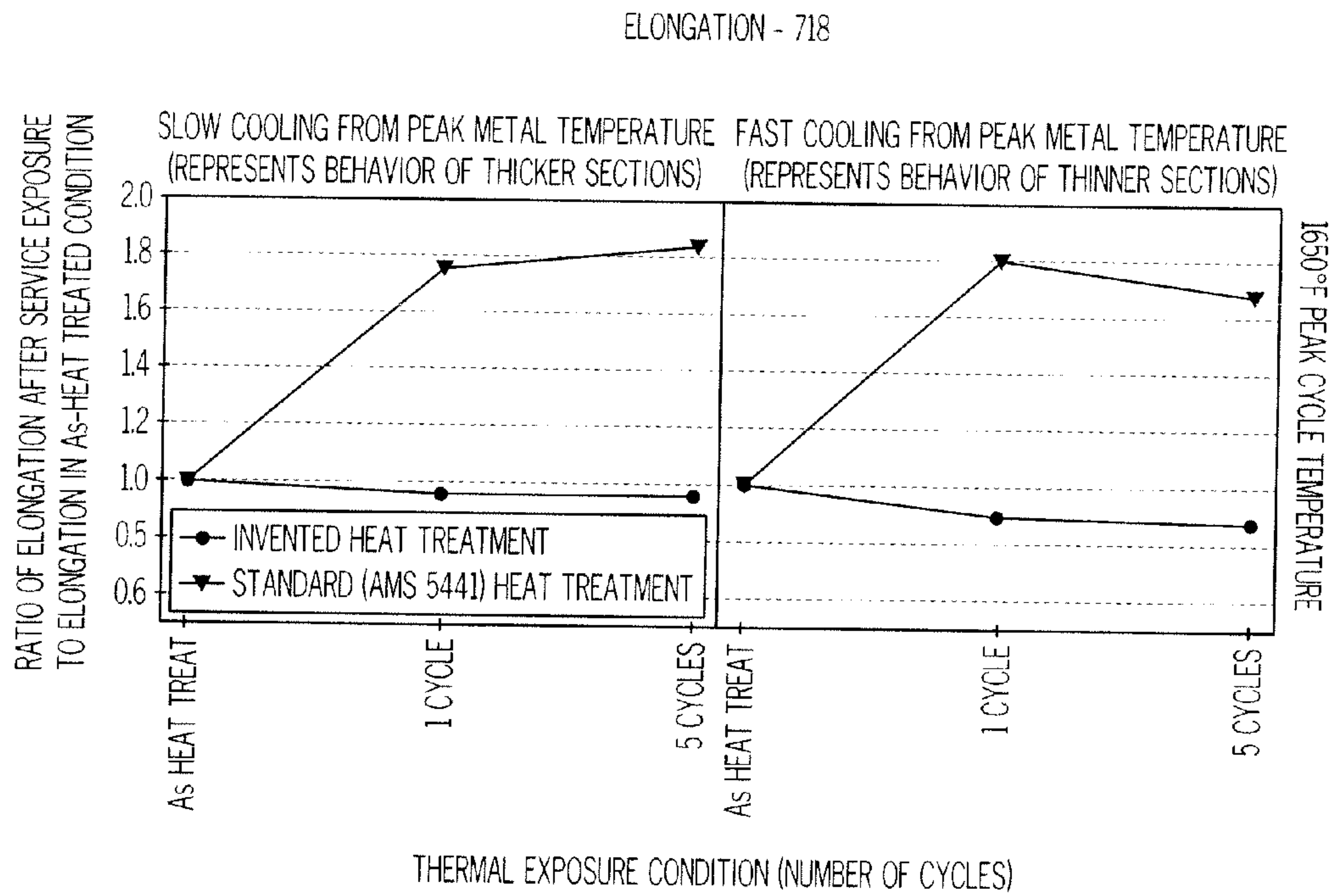


FIG. 19

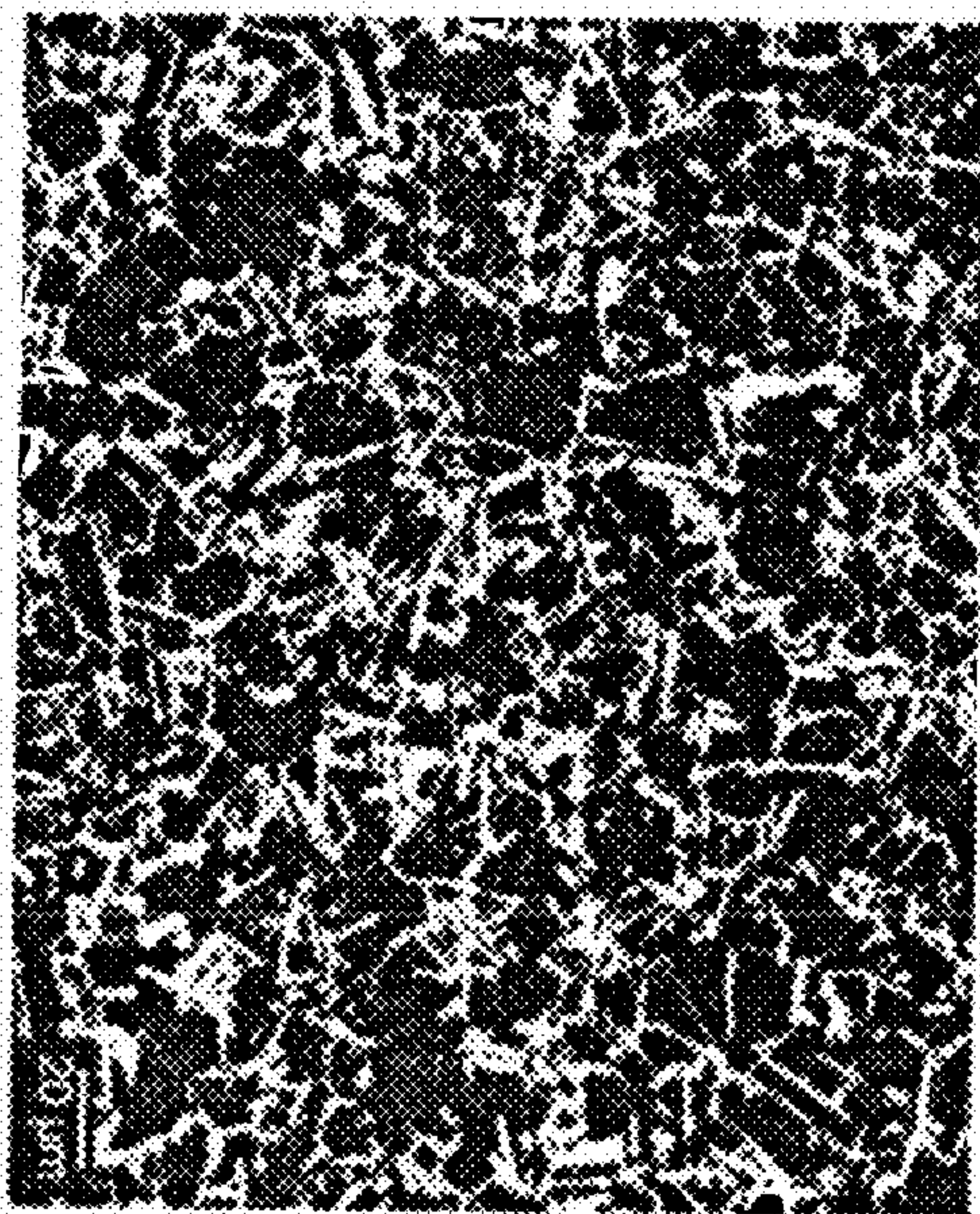


FIG. 20A

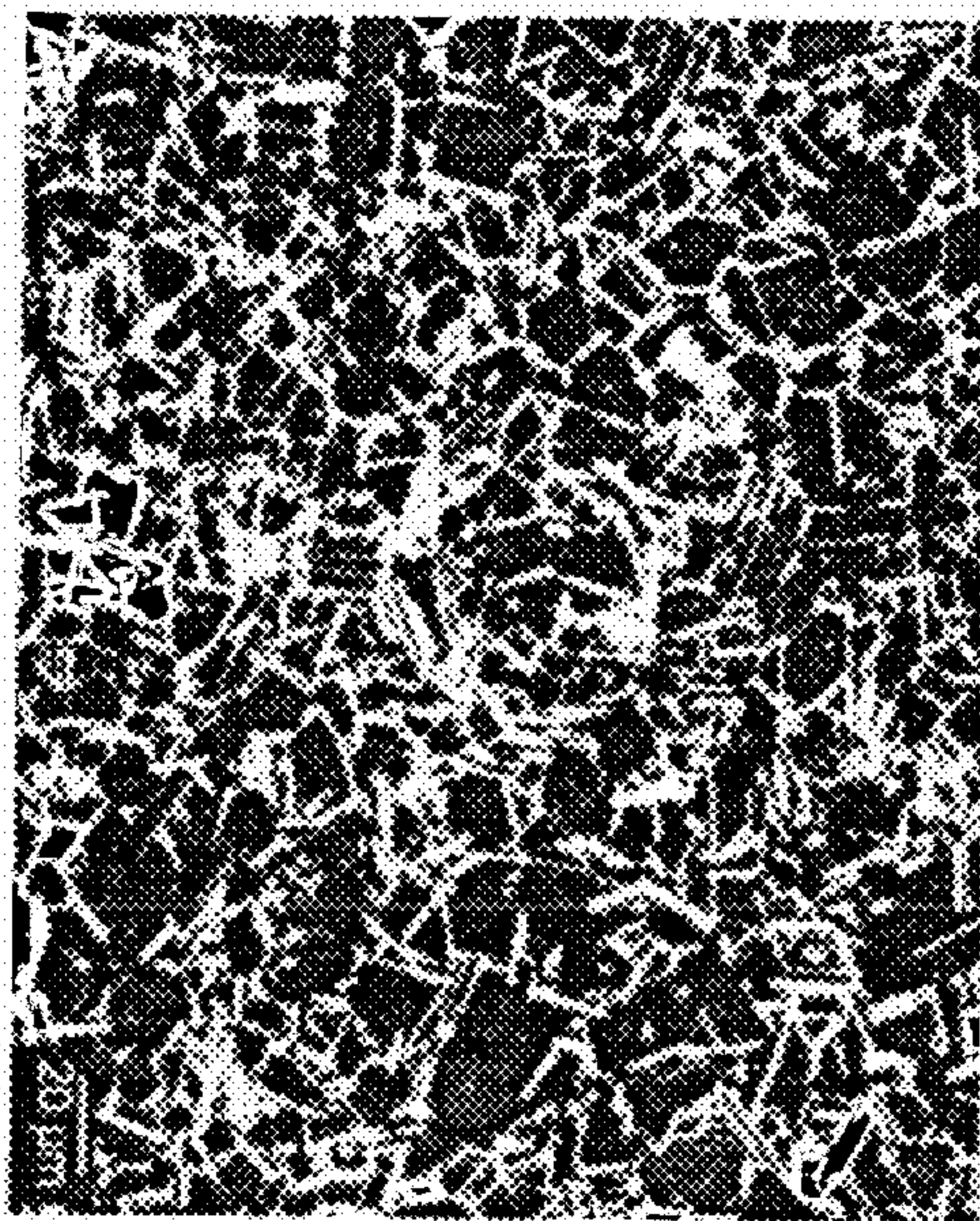


FIG. 20B

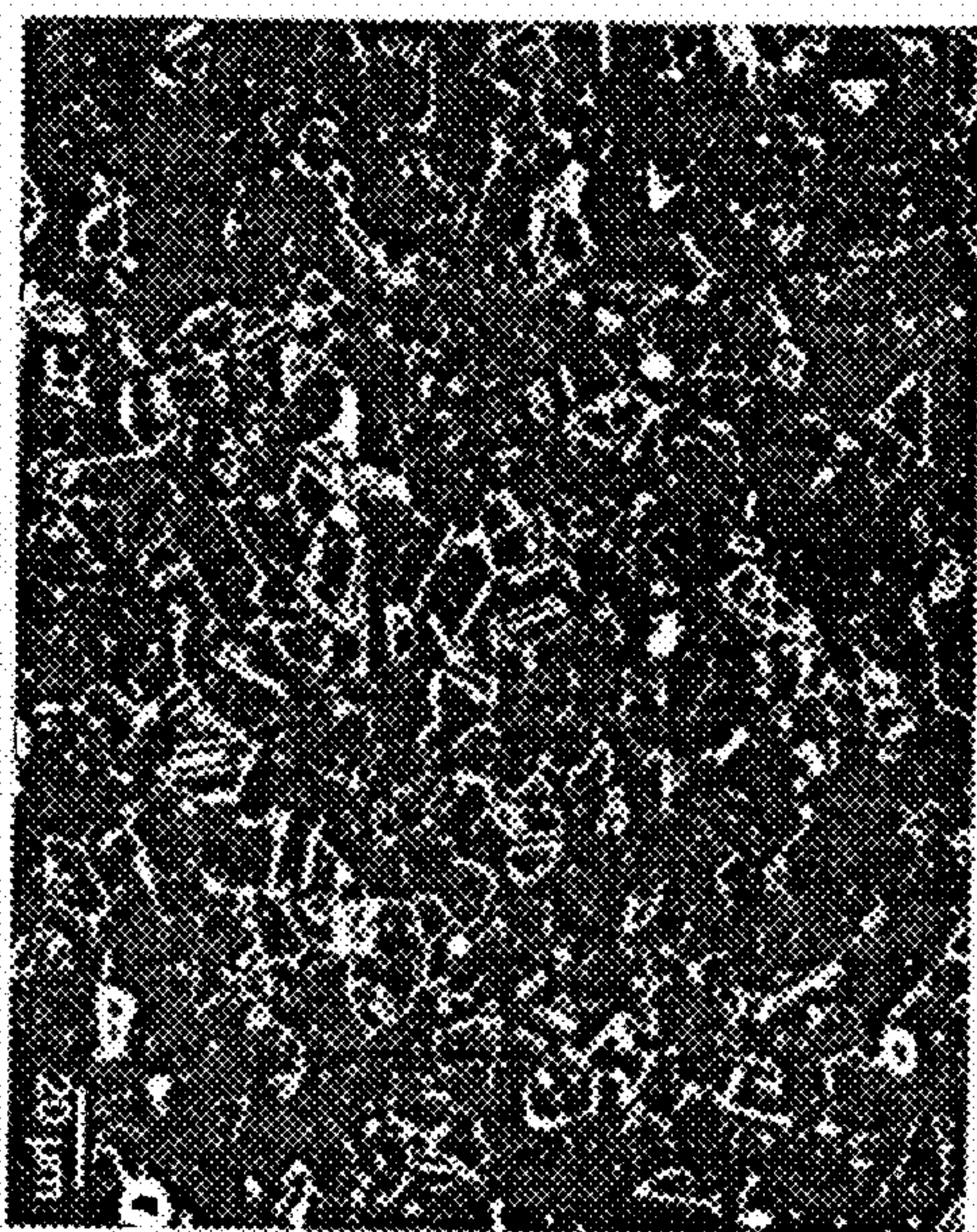


FIG. 20C
(PRIOR ART)

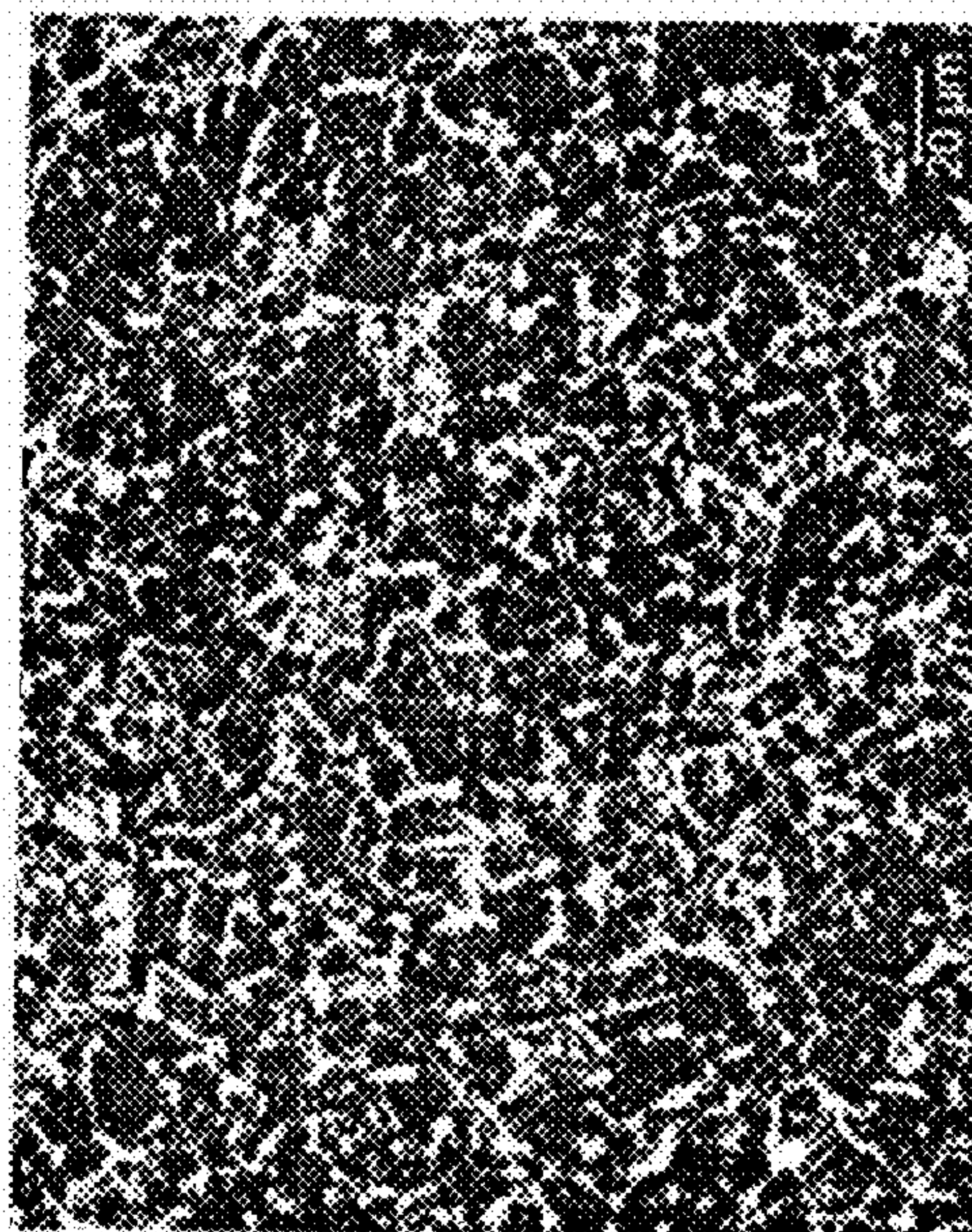


FIG. 20D
(PRIOR ART)

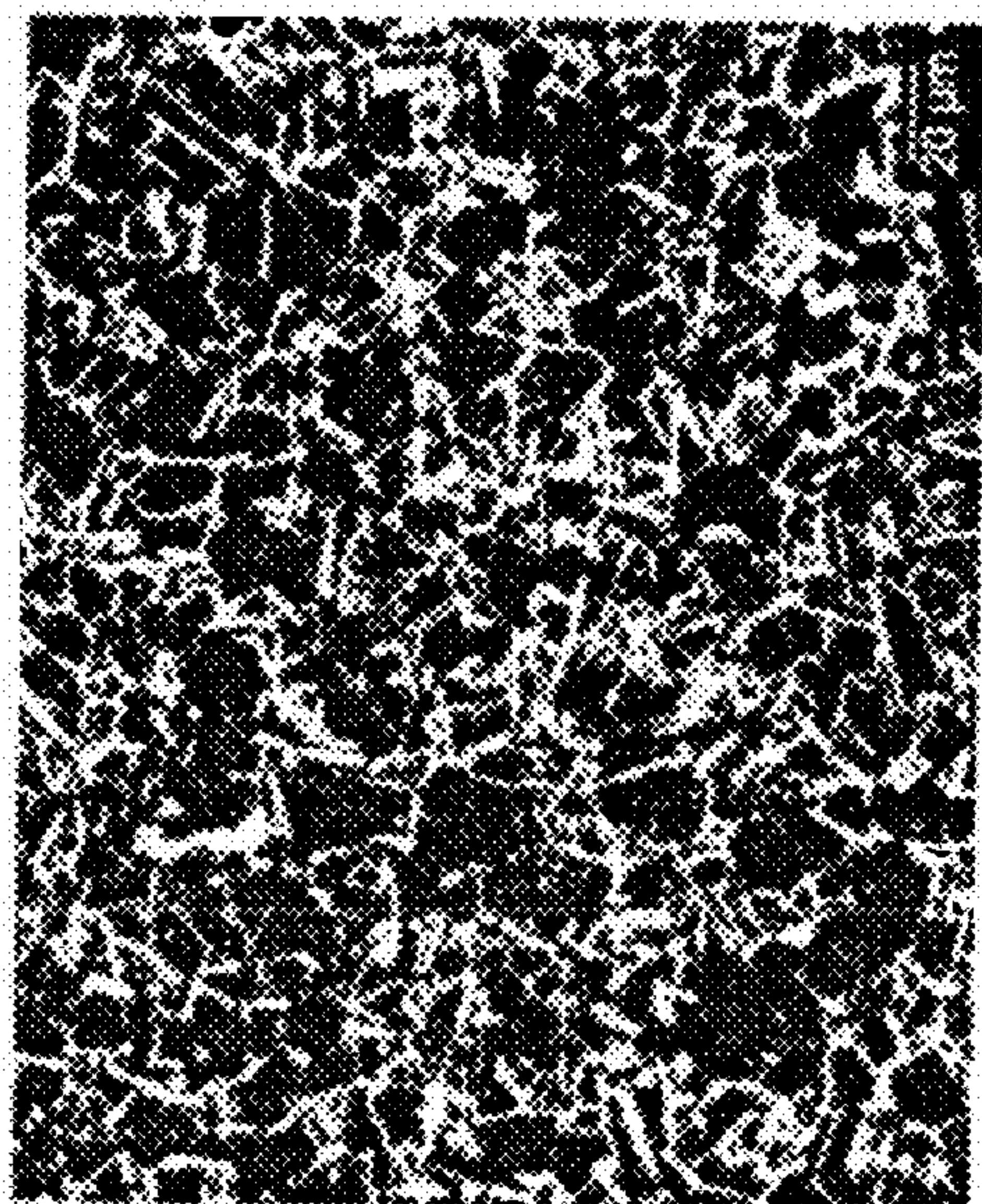


FIG. 21A

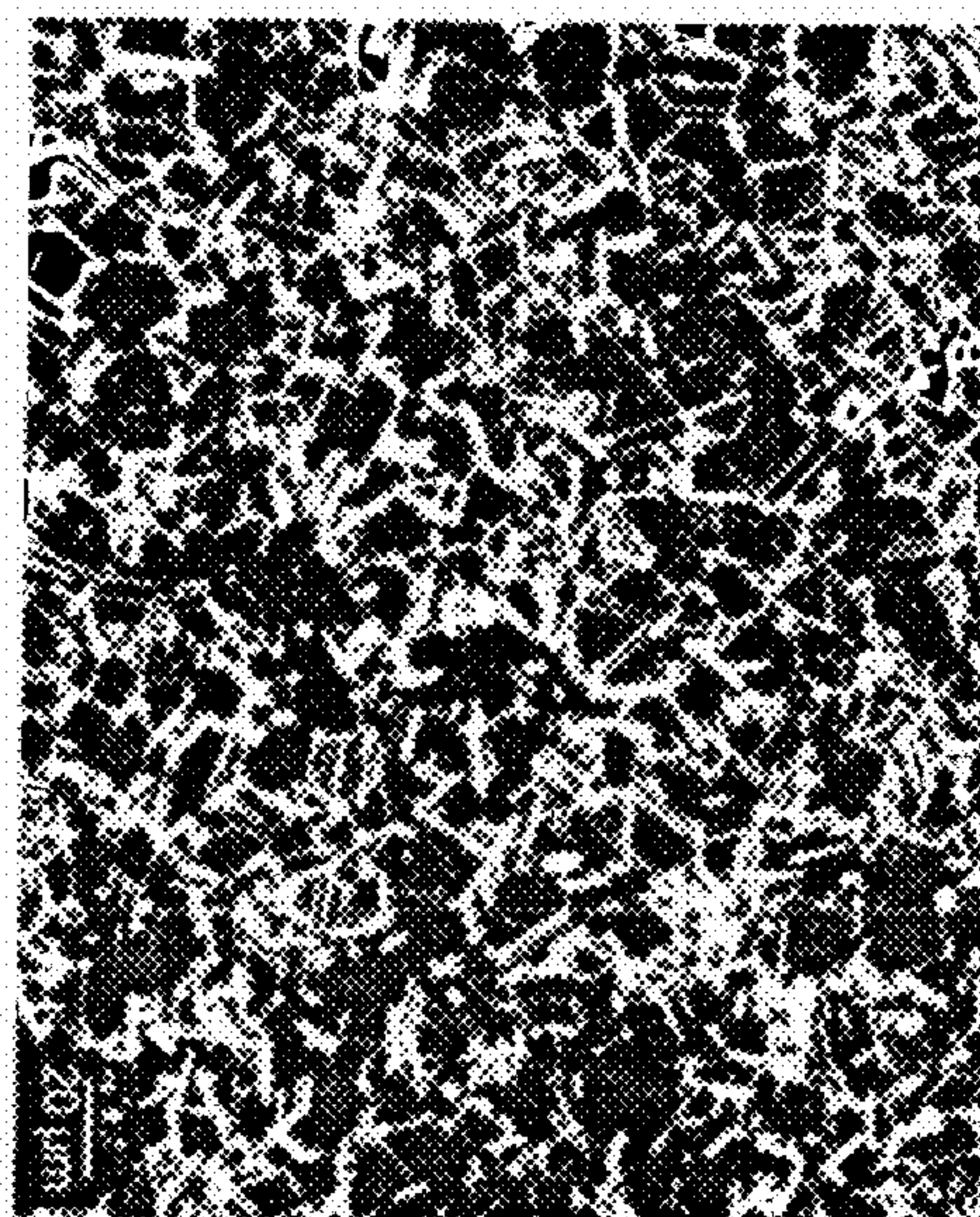


FIG. 21B

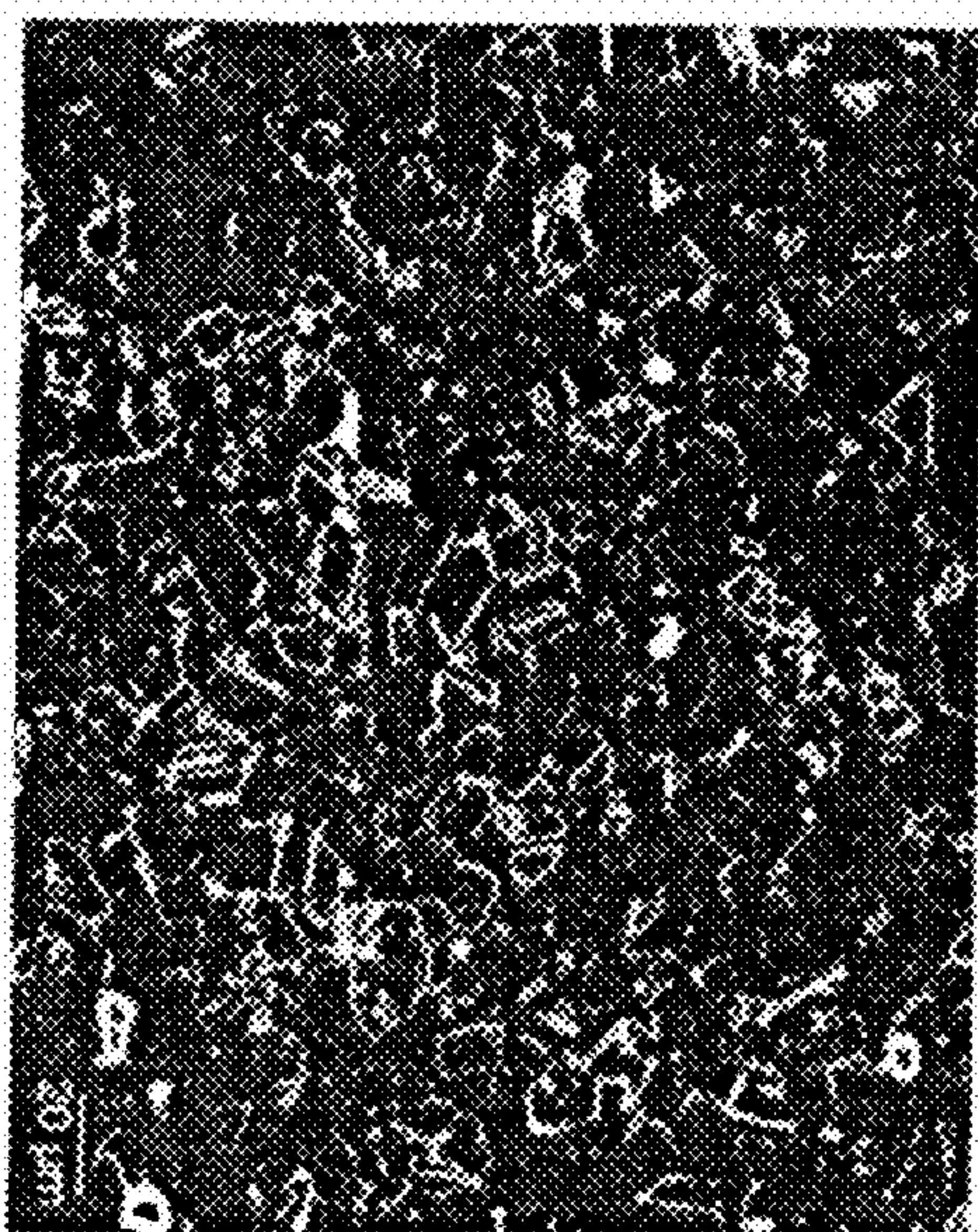


FIG. 21C
(PRIOR ART)

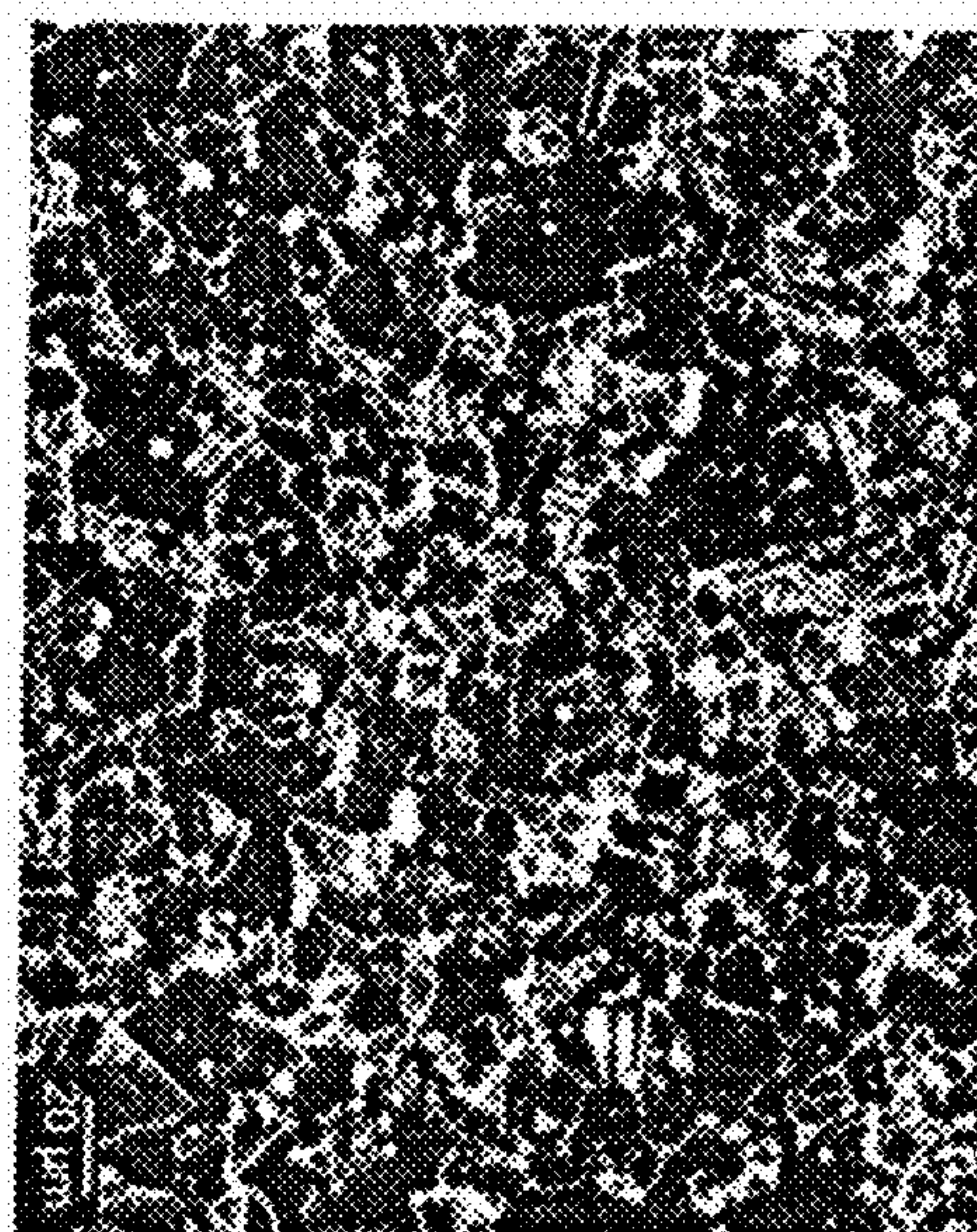


FIG. 21D
(PRIOR ART)

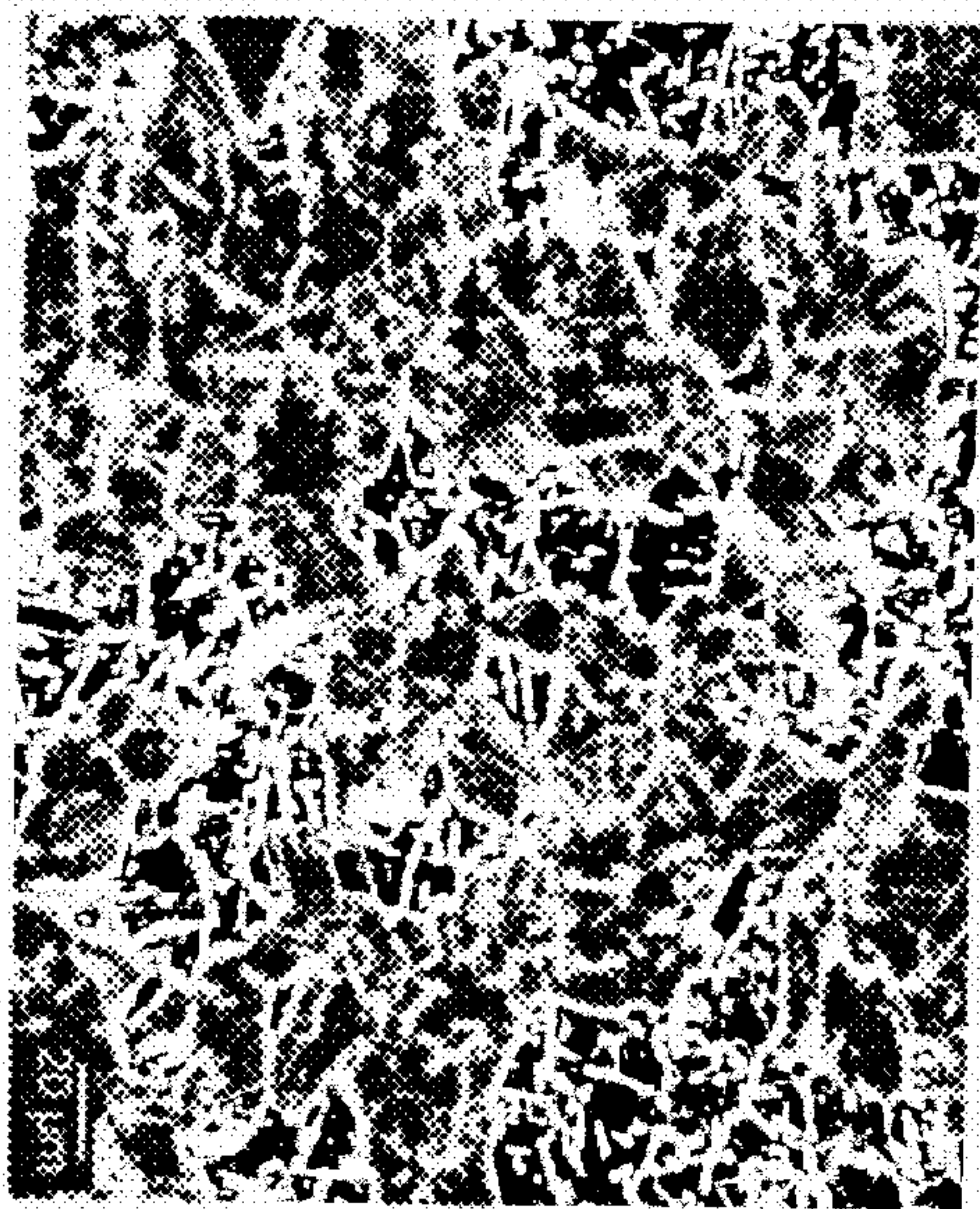


FIG. 22A

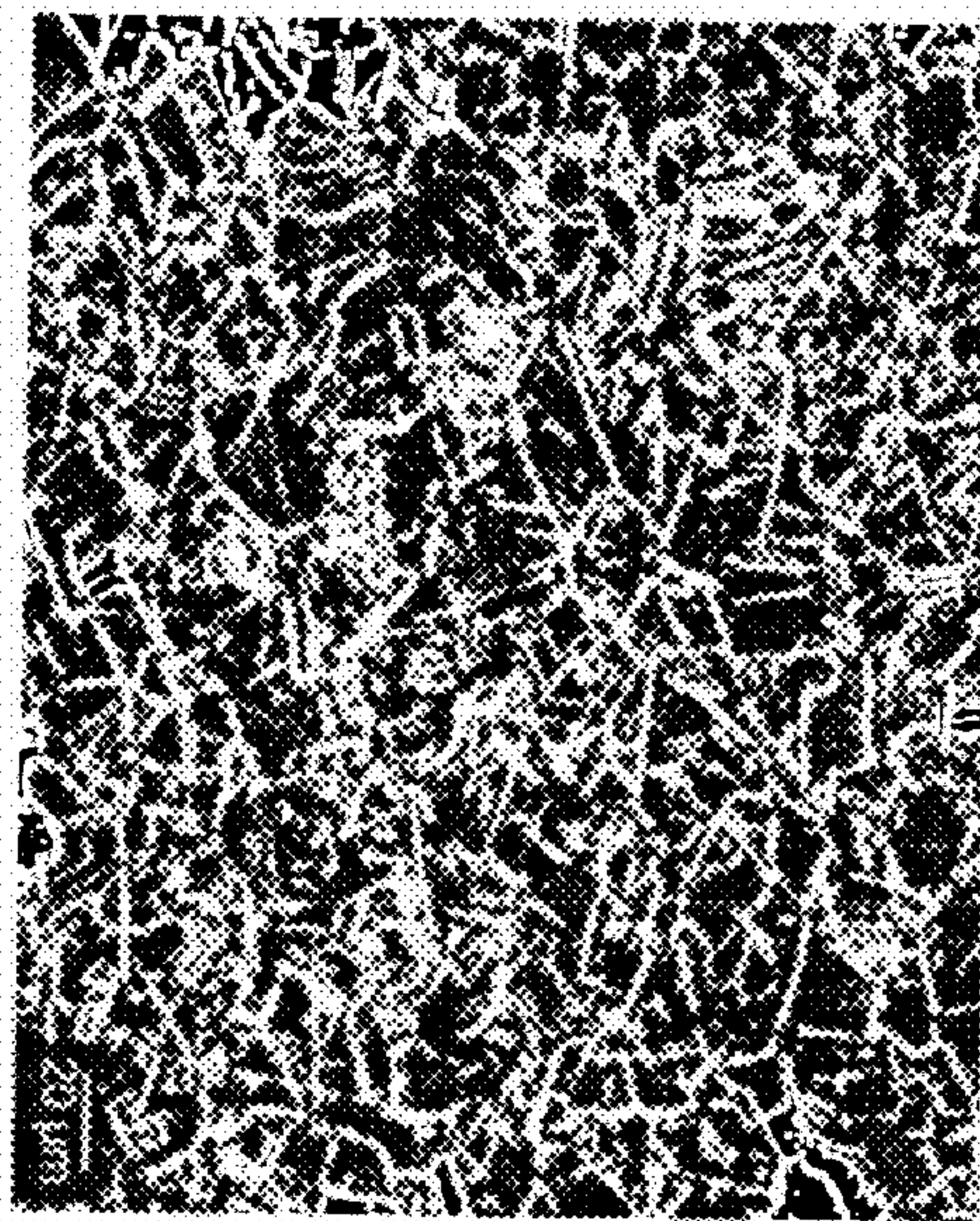


FIG. 22B

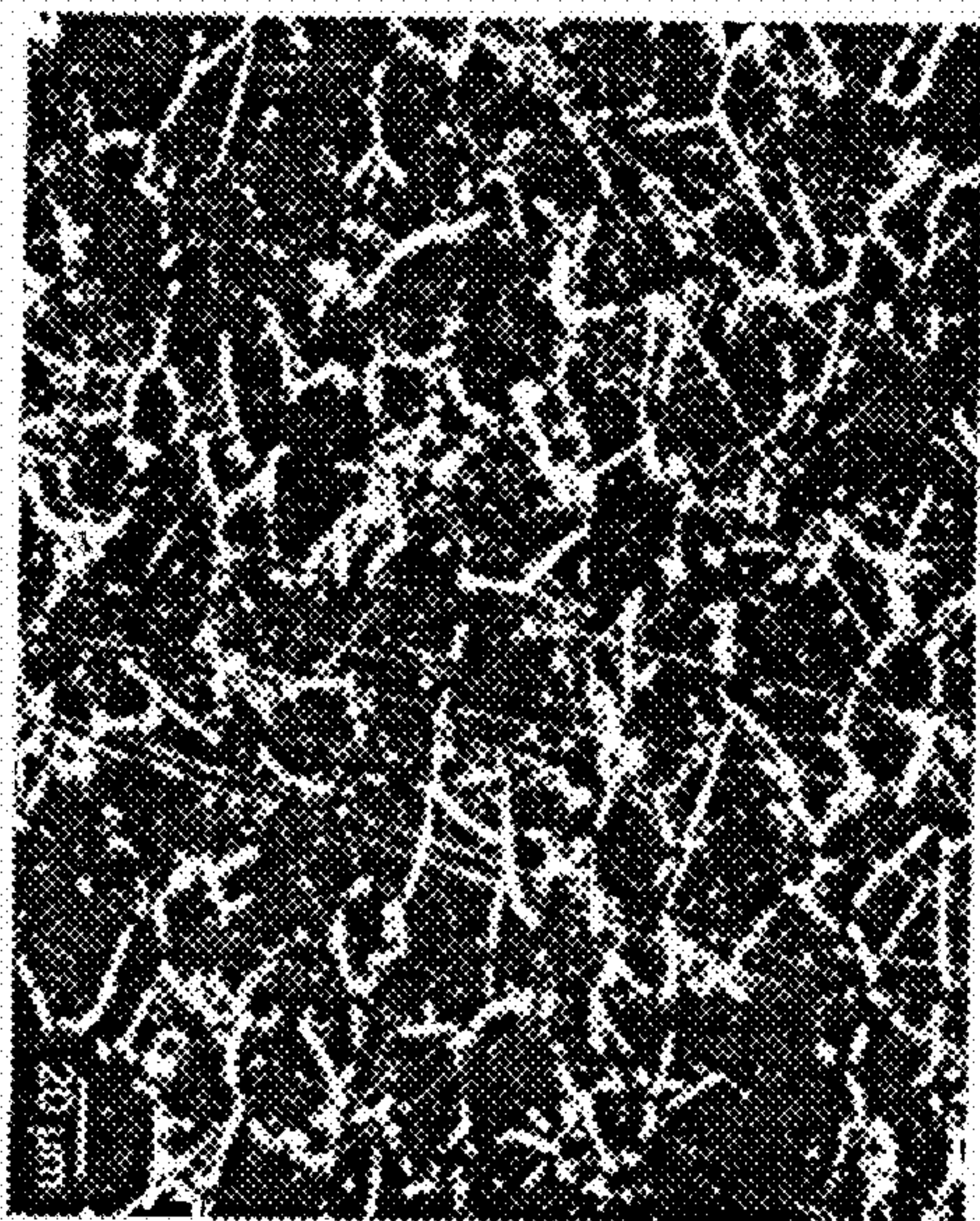


FIG. 22C
(PRIOR ART)

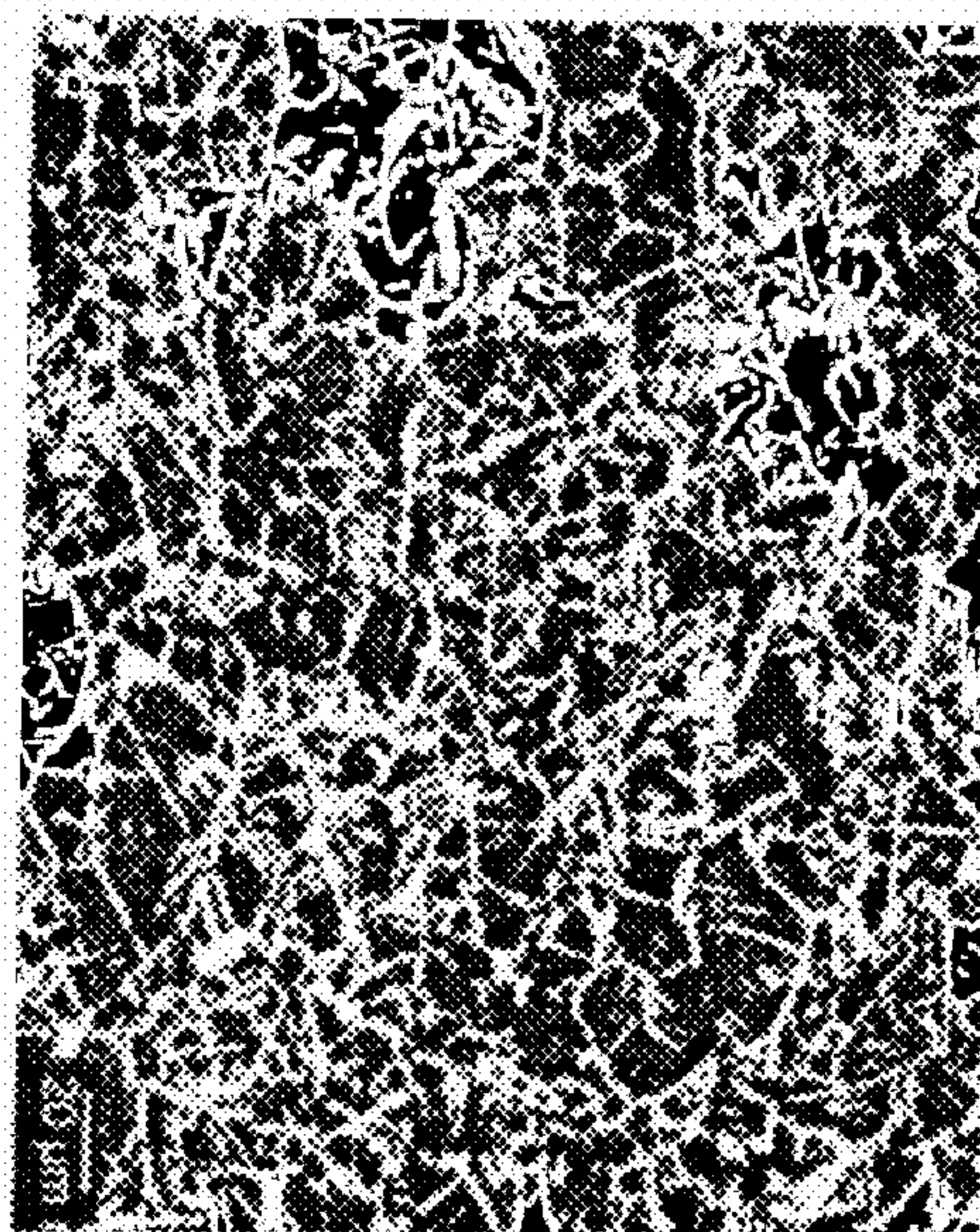


FIG. 22D
(PRIOR ART)

**NICKEL-BASE ALLOY HEAT TREATMENTS,
NICKEL-BASE ALLOYS, AND ARTICLES
INCLUDING NICKEL-BASE ALLOYS**

BACKGROUND OF THE TECHNOLOGY

[0001] 1. Field of the Technology

[0002] Embodiments of the present invention generally relate to methods of heat treating nickel-base alloys.

[0003] 2. Description of the Background of the Technology

[0004] Alloy 718 (UNS 07718) is one of the most widely used nickel-base alloys and is described generally in U.S. Pat. No. 3,046,108, the specification of which is hereby incorporated herein by reference in its entirety. Alloy 718 comprises elemental constituents within the ranges shown in the following table, plus incidental impurities.

	Element										
	Ni	Cr	C	Mn	Si	Mo	Ni + Ta	Ti	Al	B	Fe
weight %	50.0 to 55.0	17 to 21.0	up to 0.08	up to 0.35	up to 0.35	2.8 to 3.3	4.75 to 5.5	0.65 to 1.15	0.20 to 0.8	up to 0.006	rem.

[0005] The extensive use of Alloy 718 is at least partially attributable to several advantageous properties of the alloy. For example, Alloy 718 has high strength and stress-rupture properties up to about 1200° F. (648.9° C.). Additionally, Alloy 718 has good processing characteristics, such as favorable castability and hot-workability, as well as good weldability. These characteristics permit one to readily fabricate components made from Alloy 718 and, when necessary, repair those components. As discussed below, several of Alloy 718's favorable properties result from the alloy's precipitation-hardened microstructure, which is predominantly strengthened by γ'' -phase precipitates.

[0006] Precipitation-hardened, nickel-base alloys include two principal strengthening phases: γ' -phase (or "gamma prime") precipitates and γ'' -phase (or "gamma double prime") precipitates. Both the γ' -phase and the γ'' -phase are stoichiometric, nickel-rich intermetallic compounds. However, the γ' -phase typically comprises aluminum and titanium (i.e., $\text{Ni}_3(\text{Al}, \text{Ti})$) as the major alloying elements, while the γ'' -phase includes primarily niobium (i.e., Ni_3Nb). While both the γ' -phase and the γ'' -phase form coherent precipitates in the face centered cubic austenite matrix, because the misfit strain energy associated with the γ'' -phase precipitates (which have a body centered tetragonal crystal structure) is larger than that of the γ' -phase precipitates (which have a face centered cubic crystal structure), γ'' -phase precipitates tend to be more efficient strengtheners than γ' -phase precipitates. That is, for the same precipitate volume fraction and particle size, nickel-base alloys strengthened primarily by γ'' -phase precipitates are generally stronger than nickel-base alloys strengthened primarily by γ' -phase precipitates.

[0007] One disadvantage of nickel-base alloys including a γ'' -phase precipitate strengthened microstructure is that the γ'' -phase is unstable at temperatures higher than about 1200° F. (648.9° C.) and will transform into the more stable δ -phase (or "delta-phase"). While δ -phase precipitates have the same composition as γ'' -phase precipitates (i.e., Ni_3Nb), δ -phase precipitates have an orthorhombic crystal structure and are

incoherent with the austenite matrix. Accordingly, the strengthening effect of δ -phase precipitates on the matrix is generally considered to be negligible. Therefore, a result of the transformation to δ -phase is that certain mechanical properties of Alloy 718, such as stress-rupture life, deteriorate rapidly at temperatures above about 1200° F. (648.9° C.). Therefore, the use of Alloy 718 typically has been limited to applications in which the alloy is subjected to temperatures below 1200° F. (648.9° C.).

[0008] In order to form the desired precipitation-hardened microstructure, nickel-base alloys are subjected to a heat treatment or precipitation hardening process. The precipitation hardening process for a nickel-base alloy generally involves solution treating the alloy by heating the alloy at a temperature sufficient to dissolve substantially all of γ' -phase

and γ'' -phase precipitates in the alloy (i.e., a temperature near, at, or above the solvus temperature of the precipitates), cooling the alloy from the solution treating temperature, and subsequently aging the alloy in one or more aging steps. Aging is conducted at temperatures below the solvus temperature of the gamma precipitates in order to permit the desired precipitates to develop in a controlled manner.

[0009] The development of the desired microstructure in the nickel-base alloy depends upon both the alloy composition and the precipitation hardening process (i.e., the solution treating and aging processes) employed. For example, a typical precipitation hardening procedure for Alloy 718 for high temperature service involves solution treating the alloy at a temperature of 1750° F. (954.4° C.) for 1 to 2 hours, air cooling the alloy, followed by aging the alloy in a two-step aging process. The first aging step involves heating the alloy at a first aging temperature of 1325° F. (718.3° C.) for 8 hours, cooling the alloy at about 50 to 100° F. per hour (28 to 55.6° C. per hour) to a second aging temperature of 1150° F. (621.2° C.), and aging the alloy at the second aging temperature for 8 hours. Thereafter, the alloy is air cooled to room temperature. The precipitation-hardened microstructure that results after the above-described heat treatment is comprised of discrete γ' -phase and γ'' -phase precipitates, but is predominantly strengthened by the γ'' -phase precipitates with minor amounts of the γ' -phase precipitates playing a secondary strengthening role.

[0010] In an effort to increase the allowable service temperatures of nickel-base alloys, several γ' -phase strengthened nickel-base alloys have been developed. An example of such an alloy is Waspaloy nickel-base alloy (UNS N07001), which is commercially available as ATI Waspaloy alloy from ATI Allvac, Monroe, N.C. USA. Because Waspaloy nickel-base alloy includes higher levels of alloying additions, including nickel, cobalt, and molybdenum, than Alloy 718, Waspaloy alloy typically is more costly than Alloy 718. Also, because of the faster precipitation kinetics of γ' -phase precipitates rela-

tive to γ'' -phase precipitates, the hot workability and weldability of Waspaloy alloy is generally considered to be inferior to Alloy 718.

[0011] Another γ' -phase strengthened nickel-base alloy is ATI 718Plus® alloy, which is commercially available from ATI Allvac, Monroe, N.C. ATI 718Plus® alloy is disclosed in U.S. Pat. No. 6,730,264 (“the U.S. ’264 patent”), which hereby is incorporated herein by reference in its entirety. A feature of ATI 718Plus® alloy is that the alloy’s aluminum, titanium and/or niobium levels and their relative ratio are adjusted in a manner that provides a thermally stable microstructure and advantageous high-temperature mechanical properties, including substantial rupture and creep strength. The aluminum and titanium contents of ATI 718Plus® alloy, in conjunction with niobium content, results in the alloy being strengthened by γ' -phase and γ'' -phase, with γ' -phase being the predominant strengthening phase. Unlike the relatively high titanium/low aluminum composition typical of certain other nickel-base superalloys, the composition of ATI 718Plus® alloy has a relatively high ratio of atomic percent aluminum to atomic percent titanium that is believed to increase thermal stability. The thermal stability characteristics of ATI 718Plus® alloy are important for maintaining good mechanical properties, such as stress rupture properties, after long periods of exposure to high temperatures.

[0012] ATI 718Plus® alloy can be subjected to processing including solution annealing, cooling, and aging. A typical heat treatment for ATI 718Plus® alloy is depicted in FIG. 1 as a schematic representation of a time-temperature heat treatment profile. A typical heat treatment for ATI 718Plus® alloy includes a solution treatment at temperatures between 1750° F. (954.4° C.) and 1800° F. (982.2° C.) to dissolve any γ' -phase and γ'' -phase and precipitate a small amount of δ -phase. The amount of δ -phase precipitated is typically less than about half the low temperature equilibrium content. The solution treatment is followed by aging at 1450° F. (787.8° C.) for 2 to 8 hours, and then at 1300° F. (704.4° C.) for an additional 8 hours to precipitate coherent γ' -phase particles. The alloy may be further processed to an article of manufacture or into any other desired form.

[0013] Additional heat treatments for strengthening ATI 718Plus® alloy are disclosed in U.S. Pat. Nos. 7,156,932; 7,491,275; and 7,527,702, each of which is hereby incorporated herein by reference in its entirety. U.S. Pat. No. 7,531,054 (the “U.S. ’054 patent”) discloses a heat treatment for ATI 718Plus® alloy that includes direct aging. In the process of the U.S. ’054 patent, after hot working the ATI 718Plus® alloy, the alloy is rapidly and directly cooled to an aging temperature of about 1400° F. (760° C.) to prevent the precipitation of coarse γ' -phase precipitates. The cooled alloy is aged at the aging temperature or is further cooled to room temperature.

[0014] In general, precipitation hardened alloys are not designated for use above their age hardening temperatures. Precipitation hardened nickel alloys have not been used in applications where the alloy may experience thermal cycling, where the alloys may be repeatedly exposed to temperatures above their age hardening temperatures and then cooled to temperatures below their age hardening temperatures. Conventional age hardening practices for nickel-base alloys, as summarized above, would not result in consistent mechanical properties over the service period for nickel-base alloys that would be exposed to thermal cycling in which temperatures exceed the alloy’s age hardening temperature.

[0015] It would be desirable to provide a heat treatment for precipitation hardened nickel-base alloys that provides a robust microstructure and imparts properties that are not significantly affected by thermal cycling. A nickel-base alloy treated in this way may be advantageous for use in, for example, face sheet and honeycomb core of thermal protection systems for hypersonic flight vehicles, and as a material in other articles of manufacture that experience in-service thermal cycling.

SUMMARY

[0016] According to one aspect of the present disclosure, a method for heat treating a 718-type nickel-base alloy comprises heating a 718-type nickel-base alloy to a heat treating temperature, and holding the 718-type nickel-base alloy at the heat treating temperature for a heat treating time sufficient to form an equilibrium or near-equilibrium concentration of δ -phase grain boundary precipitates within the nickel-base alloy. The heat treating results in the formation of up to 25 percent by weight of total γ' -phase and γ'' -phase within the nickel-base alloy. After holding the 718-type alloy at the heat treating temperature for the heat treating time, the 718-type nickel-base alloy is cooled and retains the δ -phase grain boundary precipitates in the alloy.

[0017] According to another aspect of the present disclosure, a method of heat treating a nickel-base alloy comprises heating the nickel-base alloy to a heat treating temperature in a heat treating temperature range of a temperature that is 20° F. greater than the nose of the Time-Temperature-Transformation diagram (“TTT diagram”) for delta phase precipitation up to 100° F. (55.6° C.) below the nose of the TTT diagram, and holding the nickel-base alloy within the heat treating temperature range for a heat treating time in a range of 30 minutes to 300 minutes. After holding the nickel-base alloy within the heat treating temperature range for the heat treating time, the nickel-base alloy is air cooled to ambient temperature. In a non-limiting embodiment, the nickel-base alloy is cooled at a cooling rate no greater than 1° F. per minute (0.56° C. per minute).

[0018] In a non-limiting embodiment, the nickel-base alloy comprises, in percent by weight, 0.01 to 0.05 carbon, up to 0.35 manganese, up to 0.035 silicon, 0.004 to 0.020 phosphorus, up to 0.025 sulfur, 17.00 to 21.00 chromium, 2.50 up to 3.10 molybdenum, 5.20 up to 5.80 niobium, 0.50 up to 1.00 titanium, 1.20 to 1.70 aluminum, 8.00 to 10.00 cobalt, 8.00 to 10.00 iron, 0.008 to 1.40 tungsten, 0.003 to 0.008 boron, nickel, and incidental impurities.

[0019] According to an additional aspect of the present disclosure, a 718-type nickel-base alloy is provided comprising nickel, chromium, and iron. The nickel-base alloy is strengthened by niobium and, optionally one or more of aluminum and titanium alloying additions, and the alloy comprises an austenite matrix including austenite grain boundaries. An equilibrium or near-equilibrium concentration of δ -phase precipitates exists at the austenite grain boundaries in the 718-type alloy, and the alloy includes up to 25 percent by weight of γ' -phase and γ'' precipitates.

[0020] According to a further aspect of the present disclosure, a process for making an article of manufacture includes at least one of the methods disclosed herein. In certain non-limiting embodiments, the process may be adapted for making an article of manufacture selected from a face sheet, a honeycomb core, and a honeycomb panel of a thermal protection system for a hypersonic flight vehicle.

[0021] According to yet another aspect of the present disclosure, an article of manufacture comprises an alloy disclosed herein. Such an article of manufacture may be selected from, but is not limited to, a face sheet, a honeycomb core, and a honeycomb panel of a thermal protection system for a hypersonic flight vehicle.

BRIEF DESCRIPTION OF THE DRAWINGS

[0022] The features and advantages of alloys and methods described herein may be better understood by reference to the accompanying drawings in which:

[0023] FIG. 1 is temperature-time heat treatment diagram of a conventional prior art heat treatment for strengthening nickel-base alloys;

[0024] FIG. 2 is a schematic representation of one example of a metallic thermal protection system;

[0025] FIG. 3A is a schematic representation of one example of a honeycomb panel;

[0026] FIG. 3B is a schematic representation of an exploded view of one example of a honeycomb panel;

[0027] FIG. 4 is a flow diagram of a non-limiting embodiment of a heat treatment for a nickel-base alloy according to the present disclosure;

[0028] FIG. 5A is a Time-Temperature-Transformation curve for Alloy 718 nickel-base superalloy;

[0029] FIG. 5B is a Time-Temperature-Transformation curve for ATI 718Plus® alloy;

[0030] FIG. 6 is a schematic temperature-time plot for a non-limiting embodiment of a method according to the present disclosure for heat treating a nickel-base alloy;

[0031] FIG. 7 is a schematic representation of thermal cycling used to evaluate non-limiting embodiments of methods of heat treating nickel-base alloys according to the present disclosure;

[0032] FIG. 8 provides plots of ultimate tensile strength as a function of number of thermal cycles for ATI 718Plus® alloy treated with non-limiting heat treating methods according to the present disclosure, and compared with conventional γ'/γ'' heat treating methods before and after thermal cycling to 1650° F. (898.9° C.) and 1550° F. (843.3° C.);

[0033] FIG. 9 provides plots of relative retained ultimate tensile strength as a function of number of thermal cycles for ATI 718Plus® alloy treated with non-limiting heat treating methods according to the present disclosure, and compared with conventional γ'/γ'' heat treating methods before and after thermal cycling to 1650° F. (898.9° C.) and 1550° F. (843.3° C.);

[0034] FIG. 10 provides plots of yield strength as a function of number of thermal cycles for ATI 718Plus® alloy treated with non-limiting heat treating methods according to the present disclosure, and compared with conventional γ'/γ'' heat treating methods before and after thermal cycling to 1650° F. (898.9° C.) and 1550° F. (843.3° C.);

[0035] FIG. 11 includes plots of relative retained yield strength as a function of number of thermal cycles for ATI 718Plus® alloy treated with non-limiting heat treating methods according to the present disclosure, and compared with conventional γ'/γ'' heat treating methods before and after thermal cycling to 1650° F. (898.9° C.) and 1550° F. (843.3° C.);

[0036] FIG. 12 includes plots of percent elongation as a function of number of thermal cycles for ATI 718Plus® alloy treated with non-limiting heat treating methods according to the present disclosure, and compared with conventional γ'/γ''

heat treating methods before and after thermal cycling to 1650° F. (898.9° C.) and 1550° F. (843.3° C.);

[0037] FIG. 13 includes plots of relative percent elongation as a function of number of thermal cycles for ATI 718Plus® alloy treated with non-limiting heat treating methods according to the present disclosure, and compared with conventional γ'/γ'' heat treating methods before and after thermal cycling to 1650° F. (898.9° C.) and 1550° F. (843.3° C.);

[0038] FIG. 14 includes plots of ultimate tensile strength as a function of number of thermal cycles for Alloy 718 treated with non-limiting heat treating methods according to the present disclosure, and compared with conventional γ'/γ'' heat treating methods before and after thermal cycling to 1650° F. (898.9° C.);

[0039] FIG. 15 includes plots of relative retained ultimate tensile strength as a function of number of thermal cycles for Alloy 718 treated with non-limiting heat treating methods according to the present disclosure, and compared with conventional γ'/γ'' heat treating methods before and after thermal cycling to 1650° F. (898.9° C.);

[0040] FIG. 16 includes plots of yield strength as a function of number of thermal cycles for Alloy 718 treated with non-limiting heat treating methods according to the present disclosure, and compared with conventional γ'/γ'' heat treating methods before and after thermal cycling to 1650° F. (898.9° C.);

[0041] FIG. 17 includes plots of relative retained yield strength as a function of number of thermal cycles for Alloy 718 treated with non-limiting heat treating methods according to the present disclosure, and compared with conventional γ'/γ'' heat treating methods before and after thermal cycling to 1650° F. (898.9° C.);

[0042] FIG. 18 includes plots of percent elongation as a function of number of thermal cycles for Alloy 718 treated with non-limiting heat treating methods according to the present disclosure, and compared with conventional γ'/γ'' heat treating methods before and after thermal cycling to 1650° F. (898.9° C.);

[0043] FIG. 19 includes plots of relative percent elongation as a function of number of thermal cycles for Alloy 718 treated with non-limiting heat treating methods according to the present disclosure, and compared with conventional γ'/γ'' heat treating methods before and after thermal cycling to 1650° F. (898.9° C.);

[0044] FIG. 20A is a dark field optical micrograph of a surface region of a sheet of ATI 718Plus® alloy heat treated according to a non-limiting embodiment of the present disclosure;

[0045] FIG. 20B is a dark field optical micrograph of a surface region of a sheet of ATI 718Plus® alloy heat treated according to a non-limiting embodiment of the present disclosure after 5 thermal cycles from ambient temperature to 1650° F. (898.9° C.) and back to ambient temperature;

[0046] FIG. 20C is a dark field optical micrograph of a surface region of a sheet of ATI 718Plus® alloy heat treated according to a conventional γ'/γ'' heat treatment;

[0047] FIG. 20D is a dark field optical micrograph of a surface region of a sheet of ATI 718Plus® alloy heat treated according to a conventional γ'/γ'' heat treatment after 5 thermal cycles from ambient temperature to 1650° F. (898.9° C.) and back to ambient temperature;

[0048] FIG. 21A is a dark field optical micrograph of a surface region of a sheet of ATI 718Plus® alloy heat treated according to a non-limiting embodiment of the present disclosure;

[0049] FIG. 21B is a dark field optical micrograph of a surface region of a sheet of ATI 718Plus® alloy heat treated according to a non-limiting embodiment of the present disclosure after 5 thermal cycles from ambient temperature to 1550° F. (843.3° C.) and back to ambient temperature;

[0050] FIG. 21C is a dark field optical micrograph of a surface region of a sheet of ATI 718Plus® alloy heat treated according to a conventional γ'/γ'' heat treatment;

[0051] FIG. 21D is a dark field optical micrograph of a surface region of a sheet of ATI 718Plus® alloy heat treated according to a conventional γ'/γ'' heat treatment after 5 thermal cycles from ambient temperature to 1550° F. (843.3° C.) and back to ambient temperature;

[0052] FIG. 22A is a dark field optical micrograph of a surface region of a sheet of Alloy 718 heat treated according to a non-limiting embodiment of the present disclosure;

[0053] FIG. 22B is a dark field optical micrograph of a surface region of a sheet of Alloy 718 heat treated according to a non-limiting embodiment of the present disclosure after 5 thermal cycles from ambient temperature to 1650° F. (898.9° C.) and back to ambient temperature;

[0054] FIG. 22C is a dark field optical micrograph of a surface region of a sheet of Alloy 718 heat treated according to a conventional γ'/γ'' heat treatment; and

[0055] FIG. 22D is a dark field optical micrograph of a surface region of a sheet of Alloy 718 heat treated according to a conventional γ'/γ'' heat treatment after 5 thermal cycles from ambient temperature to 1650° F. (898.9° C.) and back to ambient temperature.

[0056] The reader will appreciate the foregoing details, as well as others, upon considering the following detailed description of certain non-limiting embodiments according to the present disclosure.

DETAILED DESCRIPTION OF CERTAIN NON-LIMITING EMBODIMENTS

[0057] In the present description of non-limiting embodiments, other than in the operating examples or where otherwise indicated, all numbers expressing quantities or characteristics are to be understood as being modified in all instances by the term “about”. At the very least, and not as an attempt to limit the application of the doctrine of equivalents to the scope of the claims, each numerical parameter should at least be construed in light of the number of reported significant digits and by applying ordinary rounding techniques.

[0058] Any patent, publication, or other disclosure material that is said to be incorporated, in whole or in part, by reference herein is incorporated herein only to the extent that the incorporated material does not conflict with existing definitions, statements, or other disclosure material set forth in the present disclosure. As such, and to the extent necessary, the disclosure as set forth herein supersedes any conflicting material incorporated herein by reference. Any material, or portion thereof, that is said to be incorporated by reference herein, but which conflicts with existing definitions, statements, or other disclosure material set forth herein is only incorporated to the extent that no conflict arises between that incorporated material and the existing disclosure material.

[0059] Certain nickel-base alloys are being considered for use as face sheet and core elements for honeycomb panels that

will be used in thermal protection systems for hypersonic flight vehicles. The surface temperature of a hypersonic flight vehicle when in service will cycle between ground temperature and about 2200° F. (1204° C.) at least once per flight mission. Exposure of age hardened nickel-base alloys to such a thermal cycle may result in a change in the volume fraction and size of precipitate phases, particularly the γ' -phase and γ'' -phase precipitates, as compared with the as-brazed and age-hardened condition of the nickel-base alloy prior to the first flight mission flown. Further, it is to be expected that different flight missions will have different thermal exposure profiles, resulting in a microstructure and mechanical properties of the age hardened nickel-base alloy that will vary based on the mission or missions flown.

[0060] A thermal protection system (TPS) protects key components of hypersonic flight vehicles and spacecraft from melting or being otherwise damaged from the heat generated at high speeds and/or during re-entry into the atmosphere. A TPS must be lightweight, reusable, and maintainable. A schematic representation of one example of a metallic TPS (10) employing honeycomb panels is presented in FIG. 2. The metallic TPS (10) may be fastened to an external reinforcing member (12) of a component such as, for example, a cryogenic fuel tank (not shown) of a hypersonic flight vehicle or space vehicle. The metallic TPS (10) may comprise, for example, metallic honeycomb panels (14) and foil encapsulated insulation (16).

[0061] One example of a honeycomb panel (20) is schematically depicted in FIG. 3A, and an exploded schematic view of honeycomb panel (20) is depicted in FIG. 3B. Honeycomb panel (20) comprises a compartmentalized honeycomb core (22) interposed between and joined to opposing face sheets (24), thereby providing multiple enclosed chambers within the panel. As used herein, the term “honeycomb panel” refers to a metallic honeycomb core interposed or sandwiched between metallic face sheets. As used herein, the terms “honeycomb” and “honeycomb core” refer to a manufactured product comprising an arrangement of generally polygonal-shaped (e.g., hexagonal-shaped) cells formed from alloy foil and which may be applied as core material interposed or sandwiched between two face sheets of a metallic material or other suitable material to provide a honeycomb panel. As used herein, the term “face sheet” refers to metallic foil, sheet, or plate that is joined to a metallic honeycomb core as generally depicted in FIG. 2 to provide a honeycomb panel. Honeycomb cores are used to form honeycomb panels by adhesively bonding, brazing, welding, or otherwise joining face sheets to the open cells of the honeycomb core. A honeycomb panel exhibits high compression and shear properties, while minimizing the weight required to achieve these properties compared with a monolithic material. Honeycomb panels are used in aerospace, marine, and ground transportation applications in order to reduce vehicle weight and reduce fuel consumption. Methods of forming honeycomb core, face sheets, and honeycomb panels are well known to persons skilled in the art and, thus, are not further described herein.

[0062] It is believed that the aerospace industry has only seriously considered the use of metallic TPSs within the past 15 years, and little attention has been given to alloys used for the face sheet and honeycomb core of aerospace panels. Generally, the precipitation hardened alloys have been avoided, and solution strengthened or oxide dispersion strengthened

alloys have been used in TPS applications because of the inherent phase instability of a precipitation hardened alloy microstructure.

[0063] Certain non-limiting embodiments of the present invention are directed to methods of heat treating nickel-base alloys to provide a microstructure that is generally stable when subjected to thermal cycling. Because the microstructure achieved by the present methods remains substantially the same during the one or more thermal cycles to which the nickel-base alloy is subjected, the mechanical properties of the nickel-base alloy will remain substantially the same at a particular temperature when the alloy is thermally cycled back to that particular temperature. For example, non-limiting embodiments of heat treating methods according to the present disclosure provide a nickel-base alloy with certain properties at 1550° F. (843.3° C.) in a second thermal cycle that are substantially the same as the properties of the same nickel-base alloy at 1550° F. (843.3° C.) in a tenth thermal cycle, but which are not the same as the mechanical properties of the nickel-base alloy at, for example, 1650° F. (898.9° C.) or at 1700° F. (926.7° C.).

[0064] It was determined that γ' -phase contributes little to the strength of low γ' -phase volume fraction alloys such as, for example, Alloy 718, at temperatures above about 1500° F. (815.6° C.). Therefore, it was determined that heat treatments designed to optimize γ' -phase are not beneficial for applications such as hypersonic flight vehicle TPSs, which may experience repeated thermal cycling between ambient temperature and temperatures up to 2200° F. (1204° C.). Heat treatments that provide a stable microstructure during such thermal cycling would be beneficial for use in thermal protection systems.

[0065] For example, a non-limiting embodiment according to the present disclosure is directed to a method of heat treating a nickel-base alloy to produce a thermally stable microstructure in a 718-type nickel-base alloy that is able to withstand thermal cycling between ambient ground temperatures and a maximum temperature of about 1450° F. (787.8° C.) to about 75° F. (42° C.) below the δ -solvus temperature. The thermally stable microstructure is a microstructure that provides the alloy with mechanical properties that do not substantially change when exposed to thermal cycles in a temperature range between ambient temperature and a maximum temperature in a range of about 1450° F. (787.8° C.) to about 75° F. (42° C.) below the δ -solvus temperature of the alloy. If in-service thermal cycling results in the exposure of the nickel-base alloy to temperatures above the heat treating temperature range according to the present disclosure, detrimental changes to the alloy's microstructure and mechanical properties may occur.

[0066] The δ -solvus temperature for Alloy 718 is about 1881° F. (1027° C.). The 5-solvus temperature for ATI 718Plus® alloy is about 1840° F. (1004° C.). The δ -solvus temperatures of other nickel-base alloys are known or can be readily determined without undue experimentation by a person having ordinary skill in the metallurgical arts.

[0067] In a non-limiting embodiment according to this disclosure, the method results in an equilibrium or near-equilibrium concentration of grain boundary δ -phase at the grain boundaries of the austenite matrix, with precipitation of up to 25 percent by weight of total γ' -phase and γ'' -phase precipitates. Given the precipitation of an equilibrium or near-equilibrium concentration of grain boundary δ -phase in embodiments according to this disclosure, embodiments of the heat

treating methods according to this disclosure are referred to herein as “ δ -phase heat treatments”.

[0068] Embodiments of the δ -phase heat treatments according to the present disclosure provide a volume fraction of δ -phase that does not substantially decrease until in-service temperatures exceed about 75° F. (42° C.) below the δ -solvus temperature. Therefore, embodiments of the δ -phase heat treatments disclosed herein promote a stable microstructure for applications in which temperatures may cycle up to a maximum temperature of about 75° F. (42° C.) below the δ -solvus temperature. The δ -phase precipitated at the grain boundaries according to methods of the present disclosure also serves the purpose of preventing grain growth, further stabilizing the microstructure. Embodiments of the δ -phase heat treatments disclosed herein result in lower strengths in nickel-base alloys below about 1500° F. (815.6° C.). However, in comparison, while in service, a conventionally heat treated 718-type nickel-base alloy part subjected to temperatures above 1500° F. (815.6° C.) would only exhibit relatively higher strength at temperatures below 1500° F. (815.6° C.) for the first thermal cycle to which the part is subjected.

[0069] Although not limiting herein, embodiments of the δ -phase heat treatments disclosed herein can be used in conjunction with nickel-base alloy compositions containing niobium (Nb), including 718-type nickel-base alloys and derivatives thereof. As used herein, the term “nickel-base alloy” refers to an alloy including predominantly nickel, along with one or more other alloying elements and incidental impurities. As used herein, the term “718-type nickel-base alloy” means a nickel-base alloy, as defined herein, comprising or consisting of nickel, chromium, iron, strengthening additions of niobium, and optionally one or both of aluminum and titanium, along with incidental impurities. Non-limiting examples of 718-type nickel-base alloys include Alloy 718 and other alloys discussed hereinbelow.

[0070] A non-limiting example of a 718-type nickel-base alloy for which non-limiting embodiments of heat treatments according to the present disclosure are believed to be particularly well suited is a nickel-base alloy including nickel, chromium, up to 14 weight percent iron, strengthening additions of niobium, optionally one or both of aluminum and titanium alloying additions, and incidental impurities. Another non-limiting example of a 718-type nickel-base alloy for which non-limiting embodiments of heat treatments according to the present disclosure are believed to be particularly well suited is a nickel-base alloy, as defined herein, including chromium, 6 up to 14 weight percent iron, strengthening additions of niobium, optionally one or more of aluminum and titanium alloying additions, and incidental impurities.

[0071] An additional non-limiting example of a 718-type nickel-base alloys with which embodiments of heat treating methods according to the present disclosure may be used is the nickel-base alloy disclosed in U.S. Pat. No. 6,730,264 (“the '264 patent”), which comprises or consists of, in percent by weight: up to 0.1 carbon; 12 to 20 chromium; up to 4 molybdenum; up to 6 tungsten; 5 to 12 cobalt; 6 to 14 iron; 4 to 8 niobium; 0.6 to 2.6 aluminum; 0.4 to 1.4 titanium; 0.003 to 0.03 phosphorus; 0.003 to 0.015 boron; nickel; and incidental impurities; wherein a sum of the weight percent of molybdenum and the weight percent of tungsten is at least 2 and not more than 8; wherein a sum of atomic percent aluminum and atomic percent titanium is from 2 to 6; wherein a ratio of atomic percent aluminum to atomic percent titanium

is at least 1.5; and wherein the sum of atomic percent aluminum and atomic percent titanium divided by atomic percent niobium is from 0.8 to 1.3. The entire disclosure of U.S. Pat. No. 6,730,264 is hereby incorporated by reference herein.

[0072] Still another non-limiting example of a 718-type nickel-base alloy with which embodiments of heat treating methods according to the present disclosure may be used is a nickel-base alloy disclosed in the U.S. '264 patent and which comprises or consists of, in percent by weight: 50 to 55 nickel; 17 to 21 chromium; 2.8 to 3.3 molybdenum; 4.7 percent to 5.5 niobium; up to 1 cobalt; 0.003 to 0.015 boron; up to 0.3 copper; up to 0.08 carbon; up to 0.35 manganese; 0.003 to 0.03 phosphorous; up to 0.015 sulfur; up to 0.35 silicon; iron; aluminum; titanium; and incidental impurities; wherein the sum of atomic percent aluminum and atomic percent titanium is from about 2 to about 6 atomic percent; wherein the ratio of atomic percent aluminum to atomic percent titanium is at least about 1.5; and wherein the sum of atomic percent of aluminum plus atomic percent of titanium divided by atomic percent of niobium is from about 0.8 to about 1.3. In certain embodiments of the alloy, the weight percent of iron is from 12 up to 20.

[0073] Yet another non-limiting example of a 718-type nickel-base alloy with which embodiments of heat treating methods according to the present disclosure may be used is ATI 718Plus® alloy (UNS N07818), which is a nickel-base alloy that is available from ATI Allvac, Monroe, N.C., USA, and that comprises or consists of, in percent by weight: 17.00 to 21.00 chromium; 2.50 to 3.10 molybdenum; 5.20 to 5.80 niobium; 0.50 to 1.00 titanium; 1.20 to 1.70 aluminum; 8.00 to 10.00 cobalt; 8.00 to 10.00 iron; 0.008 to 1.40 tungsten; 0.003 to 0.008 boron; 0.01 to 0.05 carbon; up to 0.35 manganese; up to 0.035 silicon; 0.004 to 0.020 phosphorus; up to 0.025 sulfur; nickel; and incidental impurities. AMS 5441 and AMS 5442, which relate to corrosion and heat-resistant bars, forgings, and rings, are two AMS specifications describing heat treatments conventionally used with ATI 718Plus® alloy. Each of AMS 5441 and AMS 5442 is hereby incorporated by reference herein in its entirety.

[0074] Still another non-limiting example of a 718-type nickel-base alloy with which embodiments of heat treating methods according to the present disclosure may be used is Alloy 718 (UNS N07718), the composition of which is well known in the industry. In certain non-limiting embodiments, Alloy 718 comprises or consists of, in percent by weight: 50.0 to 55.0 nickel; 17 to 21.0 chromium; up to 0.08 carbon; up to 0.35 manganese; up to 0.35 weight percent silicon; 2.8 to 3.3 molybdenum; greater than zero up to 5.5 niobium and tantalum, wherein the sum of niobium and tantalum is 4.75 to 5.5; 0.65 to 1.15 titanium; 0.20 to 0.8 aluminum; up to 0.006 boron; iron; and incidental impurities.

[0075] As used herein, the term “mechanical properties” refers to properties of an alloy relating to the elastic or inelastic reaction when force is applied to the alloy, or that involve the relationship between stress and strain that results when force is applied to the alloy. Mechanical properties, within the meaning of the present disclosure, specifically refer to tensile strength, yield strength, elongation, and stress-rupture life. As used herein, the term “thermally stable mechanical properties” refers to a condition wherein mechanical properties of an alloy do not change by more than 20% when the alloy is subjected to repeated thermal cycling between ambient ground temperature and 75° F. (41.7° C.) below the δ -solvus temperature. As used herein, the term “ambient ground tem-

perature” is defined as any temperature of the surroundings resulting from a natural terrestrial climate at ground level.

[0076] The present inventors have noted an impact of the thermal cycle peak temperature on the degree of deterioration of mechanical properties for nickel-base alloys for a given δ -phase heat treatment according to non-limiting embodiments of the present disclosure. The choice of the δ -phase heat treating temperature should be chosen to match or closely match the expected peak in-service temperature of the nickel-base alloy.

[0077] Referring now to FIG. 4, in a non-limiting embodiment according to the present disclosure, a method for δ -phase heat treating a 718-type nickel-base alloy (30) comprises: heating (32) a 718-type nickel-base alloy to a heat treating temperature in a heat treating temperature range; holding (34) the nickel-base alloy within the heat treating temperature range for a heat treating time that is sufficient to form an equilibrium or near-equilibrium concentration of δ -phase grain boundary precipitates within the nickel-base alloy, and also up to 25 percent by weight of total γ' -phase and γ'' -phase within the nickel-base alloy; and air cooling (36) the 718-type nickel-base alloy.

[0078] As used herein, the term “heat treating temperature” is defined as a temperature that results in precipitation of an equilibrium or near-equilibrium concentration of δ -phase precipitates at the grain boundary of a 718-type nickel-base alloy and up to 25 percent by weight of total γ' -phase and γ'' -phase. As used herein, the term “heat treating time” means a time sufficient to precipitate an equilibrium or near-equilibrium concentration of δ -phase precipitates at the grain boundaries of a 718-type nickel-base alloy and up to 25 percent by weight of total γ' -phase and γ'' -phase. As used herein, the term “equilibrium concentration” is defined as the maximum concentration of δ -phase precipitates that can form at the heat treating temperature according to the composition of the nickel-base alloy or 718-type nickel-base alloy. As used herein, the term “near-equilibrium concentration” means the condition wherein a nickel-base alloy includes about 5 percent to about 35 percent by weight of δ -phase at the grain boundaries. In a non-limiting embodiment, after a δ -phase heat treatment, the nickel-base alloy may include about 6 percent to about 12 percent by weight of δ -phase precipitated at the grain boundaries. Such a result is observed to be typical for Alloy 718. In another non-limiting embodiment, after a δ -phase heat treatment, the nickel-base alloy may include about 10 percent to about 25 percent by weight of δ -phase precipitated at the grain boundaries. Such a result is observed to be typical for ATI 718Plus® alloy. It is understood that the amount of δ -phase, γ' -phase, and γ'' -phase formed during a δ -phase heat treatment according to the present disclosure depends to some degree on the specific composition of the nickel-base alloy, and the amount of such phases formed may be determined readily and without undue experimentation by those having ordinary skill.

[0079] In a non-limiting embodiment, the heat treating temperature is in a heat treating temperature range having a lower limit of 20° F. (11° C.) greater than the nose of the Time-Temperature-Transformation diagram (“TTT diagram”) for δ -phase precipitation for the specific nickel-base alloy, to an upper limit that is 100° F. (55.6° C.) below the nose for δ -phase precipitation in the specific TTT diagram. A TTT diagram for a particular nickel-base alloy is a plot of temperature as a function of the logarithm of time for the alloy. TTT diagrams are used to determine when second

phase transformations, such as δ -phase, γ' -phase, and γ'' -phase transformations, begin and end during an isothermal heat treatment for a previously solution treated nickel-base alloy. A person skilled in the art understands that a particular TTT diagram is specific to a particular alloy composition. A TTT diagram for an embodiment of Alloy 718 is reproduced in FIG. 5A, and a TTT diagram for ATI 718Plus® alloy is reproduced in FIG. 5B. The curve for δ -phase precipitation in these TTT diagrams is labeled “ δ (GB)” in FIG. 5A and “ δ (Grain)” in FIG. 5B. As is understood by one having ordinary skill in the art, the “nose” of the δ -phase curve is known to a person of ordinary skill as being the portion of the δ -phase curve that is plotted to the earliest point in time on the time axis. For example the nose of δ -phase curve in FIG. 5A occurs at about 0.045 hours and about 900° C. The nose of the δ -phase curve in FIG. 5B occurs at about 0.035 hours and about 900° C. The curves shown in FIG. 5A and FIG. 5B are reproduced from Xie, et al., “TTT Diagram of a Newly Developed Nickel-Base Superalloy—Allvac 718Plus®, *Proceedings: Superalloys 718, 625, 706 and Derivatives 2005*, TMS (2005) pp. 193-202, which is hereby incorporated herein by reference. A person ordinarily skilled in the art is able to interpret and use TTT diagrams and, therefore, no further discussion concerning the use of TTT diagrams is needed herein. In addition, TTT diagrams for specific nickel-base alloys are publicly available or can be generated by a person having ordinary skill in the art without undue experimentation.

[0080] Referring to the schematic heat treating temperature-time profile (40) shown in FIG. 6, and with reference to the method steps generally shown in FIG. 4, a non-limiting embodiment of a method for heat treating a 718-type nickel-base alloy according to the present disclosure comprises heating (32) a 718-type nickel-base alloy to a heat treating temperature in a heat treating temperature range of 1700° F. (926.7° C.) to 1725° F. (940.6° C.). In a non-limiting embodiment of method, the heated 718-type nickel-base alloy is held (34) within the heat treating temperature range for a heat treating time of from 30 minutes to 300 minutes. After holding (34) at the heat treating temperature for the heat treating time, the 718-type nickel-base alloy is air cooled and retains δ -phase precipitates at the grain boundaries. According to embodiments of δ -phase heat treating method disclosed herein, the δ -phase grain boundary precipitates are primarily formed during the heating (32) and holding (34) steps. For this reason, the heating (32) and holding (34) steps may be collectively referred to as “ δ -phase aging”.

[0081] In a non-limiting embodiment, after holding the nickel-base alloy at the heat treating temperature for the heat treating time, the nickel-base alloy is air cooled from the heat treating temperature to ambient temperature. In a specific non-limiting embodiment, the nickel-base alloy is cooled at a cooling rate no greater than 1° F. per minute (0.56° C. per minute). Slow cooling is advantageous in certain non-limiting embodiments according to the present disclosure because some γ' -phase precipitation is possible in a nickel-base alloy. The small amount of γ' -phase that may precipitate during slow cooling will generally be coarse in structure and, therefore, have greater stability with respect to thermal cycling and less impact on the mechanical properties of the alloy. It is preferred to have small amounts of relatively stable γ' -phase precipitate during slow cooling than to have uncontrolled precipitation of γ' -phase during in-service thermal cycling.

[0082] Alloys processed according to any of the methods disclosed herein may be formed into mill products or other articles of manufacture. In certain non-limiting embodiments according to the present disclosure, a 718-type nickel-base alloy is processed into an article of manufacture selected from a foil, a honeycomb core, a face sheet, and a honeycomb panel by a method including an embodiment of a method disclosed herein. As used herein, the term “foil” refers to a sheet having a thickness less than 0.006 inch (0.15 mm) and any width and length. As a practical matter, the width of a foil is limited by the capacity of cold rolling equipment used to roll the alloy. In certain non-limiting embodiments of methods according to the present disclosure, alloys processed according to embodiments of the method disclosed herein may be processed into foils having a width up to 18 inches (0.46 m), up to 24 inches (0.61 m), or up to 36 inches (0.91 m).

[0083] For applications in which the maximum in-service temperature to which an alloy will be subjected is known and is about 1700° F. (926.7° C.) or less, non-limiting embodiments of a method according to the present disclosure may further include a stabilizing heat treatment subsequent to the step of cooling the nickel-base alloy from the heat treating temperature. In a non-limiting embodiment according to the present disclosure, the stabilizing heat treatment comprises heating the nickel-base alloy to a stabilizing heat treating temperature and holding the alloy at the temperature for at least 2 hours, or for at least 2 hours up to 4 hours. In non-limiting embodiments, the stabilizing heat treating temperature is the maximum in-service temperature to which the alloy will be subjected and is in a range of 1700° F. (926.7° C.) or less, or in a range of 1700° F. (926.7° C.) to 1450° F. (787.8° C.). As used herein, the term “maximum in-service temperature” refers to the maximum temperature that the particular nickel-base alloy is expected to experience when the alloy or an article including the alloy is used for its intended purpose. Subsequent to a stabilizing heat treatment according to the present disclosure, the nickel-base alloy is air cooled from the stabilizing heat treating temperature to ambient temperature. In another non-limiting embodiment, the nickel-base alloy is cooled at a cooling rate no greater than 1° F. per minute (0.56° C. per minute) from the stabilizing heat treating temperature to ambient temperature.

[0084] It is recognized that non-limiting embodiments of the δ -phase heat treatment and δ -phase aging according to this disclosure could be used on any form or shape of nickel-base alloy or 718-type nickel-base alloy. Various forms include commercial mill products such as, but not limited to, bar, rod, plate, sheet, strip, and extrusion. It is further recognized that non-limiting embodiments of the δ -phase heat treatment and δ -phase aging according to this disclosure also could be used on manufactured products such as, but not limited to, formed products, joined products, and the like comprising nickel-base alloys or 718-type nickel-base alloys.

[0085] In a non-limiting embodiment of a method of heat treating a nickel-base alloy according to the present disclosure, the nickel-base alloy comprises or consists of, in percent by weight: 17.00 to 21.00 chromium; 2.50 to 3.10 molybdenum; 5.20 to 5.80 niobium; 0.50 to 1.00 titanium; 1.20 to 1.70 aluminum; 8.00 to 10.00 cobalt; 8.00 to 10.00 iron; 0.008 to 1.40 tungsten; 0.003 to 0.008 boron; 0.01 to 0.05 carbon; up to 0.35 manganese; up to 0.035 silicon; 0.004 to 0.020 phosphorus; up to 0.025 sulfur; nickel; and incidental impurities. Such non-limiting embodiment further comprises: heat treating the nickel-base alloy to a heat treating temperature in a

range of 1700° F. (926.7° C.) to 1725° F. (940.6° C.); holding the nickel-base alloy at the heat treating temperature for a heat treating time in a range of 30 minutes to 300 minutes that is sufficient to form an equilibrium or near-equilibrium concentration of δ -phase grain boundary precipitates within the nickel-base alloy and up to 25 percent by weight of total γ' -phase and γ'' -phase within the alloy; and air cooling the nickel-base alloy. In non-limiting embodiments, the nickel-base alloy comprises one of a foil, a honeycomb core, a face sheet, and a honeycomb panel.

[0086] A non-limiting aspect according to the present disclosure is directed to a 718-type nickel-base alloy, as that term is defined herein, and that comprises an austenite matrix comprising grain boundaries. An equilibrium or near-equilibrium concentration of δ -phase precipitates is present at the grain boundaries, and up to 25 percent by weight of total γ' -phase and γ'' -phase is present in the alloy.

[0087] One specific non-limiting example of a 718-type nickel-base alloy according to the present disclosure comprises an austenite matrix including grain boundaries, an equilibrium or near-equilibrium concentration of δ -phase precipitates at the grain boundaries, up to 25 percent by weight of total γ' -phase and γ'' -phase precipitates, and up to 14 weight percent iron. Another specific non-limiting example of a 718-type nickel-base alloy according to the present disclosure comprises an austenite matrix including grain boundaries, an equilibrium or near-equilibrium concentration of δ -phase precipitates at the grain boundaries, up to 25 percent by weight of total γ' -phase and γ'' -phase precipitates, and 6 weight percent up to 14 weight percent iron.

[0088] Another specific, non-limiting example of a 718-type nickel-base alloy according to the present disclosure comprises an austenite matrix including grain boundaries, a near-equilibrium concentration of δ -phase precipitates at the grain boundaries, and up to 25 percent by weight of total γ' -phase and γ'' -phase precipitates. The alloy further comprises or consists of, in percent by weight: up to 0.1 carbon; 12 to 20 chromium; up to 4 molybdenum; up to 6 tungsten; 5 to 12 cobalt; 6 to 14 iron; 4 to 8 niobium; 0.6 to 2.6 aluminum; 0.4 to 1.4 titanium; 0.003 to 0.03 phosphorus; 0.003 to 0.015 boron; nickel; and incidental impurities; wherein a sum of the weight percent of molybdenum and the weight percent of tungsten is at least 2 and not more than 8; a sum of atomic percent aluminum and atomic percent titanium is from 2 to 6; a ratio of atomic percent aluminum to atomic percent titanium is at least 1.5; and the sum of atomic percent aluminum and atomic percent titanium divided by atomic percent niobium is from 0.8 to 1.3.

[0089] Yet another specific, non-limiting example of a 718-type nickel-base alloy according to the present disclosure comprises an austenite matrix including grain boundaries, a near-equilibrium concentration of δ -phase precipitates at the grain boundaries, and up to 25 percent by weight of total γ' -phase and γ'' -phase precipitates. The alloy further comprises or consists of, in percent by weight: 0 to about 0.08 carbon; 0 to about 0.35 manganese; about 0.003 to about 0.03 phosphorus; 0 to about 0.015 sulfur; 0 to about 0.35 silicon; about 17 to about 21 chromium; about 50 to about 55 nickel; about 2.8 to about 3.3 molybdenum; about 4.7 to about 5.5 niobium; 0 to about 1 cobalt; 0.003 to about 0.015 boron; 0 to about 0.3 copper; and balance iron (typically about 12 to about 20 percent), aluminum, titanium, and incidental impurities; wherein the sum of atomic percent aluminum and atomic percent titanium is from about 2 to about 6 percent; the

ratio of atomic percent aluminum to atomic percent titanium is at least about 1.5; and the sum of atomic percent of aluminum plus atomic percent titanium divided by atomic percent niobium equals from about 0.8 to about 1.3.

[0090] A further specific non-limiting example of a 718-type nickel-base alloy according to the present disclosure comprises an austenite matrix comprising grain boundaries, a near-equilibrium concentration of δ -phase precipitates at the grain boundaries, and up to 25 percent by weight of total γ' -phase and γ'' -phase precipitates. The alloy further comprises or consists of, in percent by weight: 0.01 to 0.05 carbon; up to 0.35 manganese; up to 0.035 silicon; 0.004 to 0.020 phosphorus; up to 0.025 sulfur; 17.00 to 21.00 chromium; 2.50 to 3.10 molybdenum; 5.20 up to 5.80 niobium; 0.50 up to 1.00 titanium; 1.20 to 1.70 aluminum; 8.00 to 10.00 cobalt; 8.00 to 10.00 iron; 0.008 to 1.40 tungsten; 0.003 to 0.008 boron; nickel; and incidental impurities.

[0091] Still a further non-limiting example of a 718-type nickel-base alloy according to the present disclosure comprises an austenite matrix comprising grain boundaries, a near-equilibrium concentration of δ -phase precipitates at the grain boundaries, and up to 25 percent by weight of total γ' -phase and γ'' -phase precipitates. The alloy further comprises or consists of, in percent by weight: 50.0 to 55.0 nickel; from 17 to 21.0 chromium; up to 0.08 carbon; up to 0.35 manganese; up to 0.35 silicon; from 2.8 to 3.3 molybdenum; greater than 0 up to 5.5 niobium and tantalum, wherein the sum of niobium and tantalum is from 4.75 to 5.5; from 0.65 to 1.15 titanium; from 0.20 to 0.8 aluminum; up to 0.006 boron; iron; and incidental impurities.

[0092] An aspect of this disclosure includes an article of manufacture fabricated according to a method of this disclosure and/or including an alloy according to this disclosure. Non-limiting examples of articles of manufacture according to this disclosure include a face sheet, a honeycomb core, and a honeycomb panel of a TPS for a hypersonic flight vehicle or a space vehicle.

[0093] The examples that follow are intended to further describe certain non-limiting embodiments, without restricting the scope of the present invention. Persons having ordinary skill in the art will appreciate that variations of the following examples are possible within the scope of the invention, which is defined solely by the claims.

Example 1

[0094] A sheet of a 0.080 inch (2.03 mm) thick ATI 718Plus® alloy and a 0.4 inch (10.2 mm) diameter rod of Alloy 718 were heat treated according to a non-limiting embodiment of the present disclosure by heating the two alloys to 1725° F. (940.6° C.) and holding at temperature for 3 hours. The samples were then air cooled.

[0095] For comparison purposes, samples of the same alloys were heat treated according to the following standard γ'/γ'' aging heat treatments.

[0096] A 0.080 inch (2.03 mm) thick sheet of ATI 718Plus® alloy was heated to 1750° F. (954.4° C.), held at temperature for 45 minutes, and air cooled. After cooling, the sample was aged at 1450° F. (787.8° C.) for 8 hours. The sample was cooled at 100° F./h (55.6° C./h) to 1300° F. (704.4° C.), and held at 1300° F. (704.4° C.) for 8 hours. After aging, the ATI 718Plus® alloy sample was air cooled.

[0097] In addition, a 0.4 inch (10.2 mm) diameter rod of Alloy 718 was heated to 1750° F. (954.4° C.), held at temperature for 45 minutes, and air cooled. After cooling, the

Alloy 718 sample was aged at 1325° F. (718.3° C.) for 8 hours. The sample was cooled at 100° F./h (55.6° C./h) to 1150° F. (621.1° C.), and held at 1150° F. (621.1° C.) for 8 hours. After aging, the sample was air cooled.

Example 2

[0098] The heat treated samples from Example 1 were subjected to thermal cycling. The ATI 718Plus® alloy samples were cycled from ambient temperature to either 1650° F. (898.9° or 1550° F. (843.3° C.). The Alloy 718 samples were cycled from ambient temperature to 1650° F. (898.9°. FIG. 7 is a schematic representation of the thermal cycles used, wherein the indicated temperatures are of the alloy samples, rather than the furnace temperature. The top plot included in FIG. 7 reflects a slower alloy cooling rate (about 10° F./min (5.6° C./min)) and represents the general behavior of thicker samples. The bottom plot reflects a faster cooling rate (about 1500° F./min (833° C./min)) and represents the general behavior of thinner samples. The cooling rates depicted in FIG. 7 are estimated, but the peak temperatures and hold times in FIG. 7 accurately represent what the alloys experienced.

Example 3

[0099] After exposure to thermal cycling, the samples were tensile tested at room temperature according to standard test procedures described in ASTM E8-09/E8M-09. Plots of ultimate tensile strengths of as-heat treated samples and after 1 and 5 thermal cycles are provided in FIG. 8. The plots on the left side of FIG. 8 show ultimate tensile strengths as a function of the number of thermal treatment cycles for ATI 718Plus® alloy samples that was cooled at the slower cooling rate discussed in Example 2. The plots on the right side of FIG. 8 show ultimate tensile strengths as a function of the number of thermal treatment cycles for ATI 718Plus® alloy that was cooled at the faster cooling rate discussed in Example 2. The top row of plots in FIG. 8 are for ATI 718Plus® alloy that was heat treated according to embodiments of the present disclosure as described in Example 1, thermally cycled to a peak sample temperature of 1650° F. (898.9° C.). The bottom row of plots in FIG. 8 are for ATI 718Plus® alloy that was heat treated according to embodiments of the present disclosure as described in Example 1, thermally cycled to a peak sample temperature of 1550° F. (843.3° C.).

[0100] Examination of FIG. 8 shows that the inventive δ -phase aging treatments may provide lower initial strengths than conventional γ'/γ'' aging treatments, but there is significantly less variability of ultimate tensile strengths during thermal cycling. This is more evident in FIG. 9, which displays the data of FIG. 8 but wherein the y-axis represents the ratio of ultimate tensile strength after the sample was subjected to thermal cycling to the ultimate tensile strength in the as-heat treated condition. FIG. 9 clearly shows that the δ -phase heat treatment embodiment according to the present disclosure produced an alloy exhibiting a significantly more stable ultimate tensile strength after thermal cycling for at least 5 thermal cycles.

[0101] FIG. 10 includes plots of yield strengths for the samples included in FIG. 8. The plots of FIG. 10 are in the same orientations as in FIG. 8 with regard to cooling rates and peak sample temperatures. Considering what is shown in FIG. 10, it will be seen that the inventive δ -phase aging treatments may provide lower initial yield strengths than con-

ventional γ'/γ'' aging treatments, but with significantly less variability of yield strengths for the δ -phase heat treated alloys during thermal cycling. This is more evident in FIG. 11, which displays the data of FIG. 10 but wherein the y-axis represents the ratio of yield strength after the sample was subjected to thermal cycling to the yield strength in the as-heat treated condition. FIG. 11 clearly shows that the δ -phase heat treatment embodiment according to the present disclosure produced an alloy exhibiting significantly more stable yield strength after thermal cycling for at least 5 thermal cycles.

[0102] FIG. 12 includes plots of percent elongation for the samples included in FIG. 8. The plots of FIG. 12 are in the same orientations as in FIG. 8 with regard to cooling rates and peak sample temperatures. Considering what is shown in FIG. 12, it will be seen that the inventive δ -phase aging treatments may provide higher percent elongation than conventional γ'/γ'' aging treatments, but with significantly less variability of percent elongation for the δ -phase heat treated alloys during thermal cycling. This is more evident in FIG. 13, which displays the data of FIG. 12 but wherein the y-axis represents the ratio of percent elongation after the sample was subjected to thermal cycling to the percent elongation in the as-heat treated condition. FIG. 13 clearly shows that the δ -phase heat treatment embodiment according to the present disclosure produced an alloy exhibiting a significantly more stable percent elongation after thermal cycling for at least 5 thermal cycles.

[0103] Samples of Alloy 718 as heat treated in Example 1 and as thermally cycled to 1650° F. (898.9° in Example 2) were tensile tested at room temperature according to standard test procedures described in ASTM E8-09/E8M-09. Plots of ultimate tensile strengths of as-heat treated samples and for samples after 1 and 5 thermal cycles are plotted in FIG. 14. The plots on the left side of FIG. 14 show ultimate tensile strengths as a function of the number of thermal treatment cycles for Alloy 718 alloy that was thermally cycled using the slower cooling rate described in Example 2, and the plots on the right were thermally cycled using the faster cooling rate described in Example 2.

[0104] Examination of FIG. 14 shows that the inventive δ -phase aging treatments may provide an alloy exhibiting lower initial strengths than conventional γ'/γ'' aging treatments, but also exhibiting significantly less variability in ultimate tensile strength when subjected to thermal cycling. This is more evident in FIG. 15, which displays the data of FIG. 14 but wherein the y-axis represents the ratio of ultimate tensile strength after the sample was subjected to thermal cycling to the ultimate tensile strength in the as-heat treated condition. FIG. 15 clearly shows that the δ -phase heat treatment embodiment according to the present disclosure produced an alloy exhibiting a significantly more stable ultimate tensile strength after thermal cycling for at least 5 thermal cycles.

[0105] FIG. 16 includes plots of yield strengths for the samples included in FIG. 14. The plots of FIG. 16 are in the same orientations as in FIG. 14 with regard to cooling rates and peak sample temperatures. Considering what is shown in FIG. 16, it will be seen that the inventive δ -phase aging treatments may provide lower initial yield strengths than conventional γ'/γ'' aging treatments, but with significantly less variability of yield strengths for the δ -phase heat treated alloys during thermal cycling. This is more evident in FIG. 17, which displays the data of FIG. 16 but wherein the y-axis

represents the ratio of yield strength after the sample was subjected to thermal cycling to the yield strength in the as-heat treated condition. FIG. 17 clearly shows that the δ -phase heat treatment embodiment according to the present disclosure produced an alloy exhibiting significantly more stable yield strength after thermal cycling for at least 5 thermal cycles.

[0106] FIG. 18 includes plots of percent elongation for the samples included in FIG. 14. The plots of FIG. 18 are in the same orientations as in FIG. 14 with regard to cooling rates and peak sample temperatures. Considering what is shown in FIG. 18, it will be seen that the inventive δ -phase aging treatments may provide higher percent elongation than conventional γ'/γ aging treatments, but with significantly less variability of percent elongation for the δ -phase heat treated alloys during thermal cycling. This is more evident in FIG. 19, which displays the data of FIG. 18 but wherein the y-axis represents the ratio of percent elongation after the sample was subjected to thermal cycling to the percent elongation in the as-heat treated condition. FIG. 19 clearly shows that the δ -phase heat treatment embodiment according to the present disclosure produced an alloy exhibiting a significantly more stable percent elongation after thermal cycling for at least 5 thermal cycles.

Example 4

[0107] Surface regions of the samples tensile tested in Example 3 were examined using dark field optical microscopy. FIG. 20A is a photomicrograph of a surface region of an ATI 718Plus® alloy sample that was δ -phase heat treated as described in Example 1. The thicker white platelets primarily disposed on grain boundaries in FIG. 20A are δ -phase platelets that result from the δ -phase heat treatment according to non-limiting embodiments of the present disclosure. FIG. 20B is a photomicrograph of a surface region of the same ATI 718Plus® alloy sample after being subjected to 5 thermal cycles to a peak sample temperature of 1650° F.). (898.9°. It may be seen that there is little, if any, difference in the amount of δ -phase platelets in the samples after 5 thermal cycles to 1650° F.). (898.9° peak sample temperature. This correlates well with the tensile tests of Example 3 showing that ATI 718Plus® alloy samples δ -phase heat treated as described in Example 1 exhibited a lower variability of tensile properties on thermal cycling.

[0108] FIG. 20C is a photomicrograph of a surface region of an ATI 718Plus® alloy sample that was heat treated according to the conventional γ'/γ heat treatment described in Example 1. It is observed that the microstructure includes a small amount of δ -phase grain boundary precipitates and that the amount is less than in the samples subjected to the δ -phase heat treatment, as seen in FIG. 20A. However, it is seen in FIG. 20D that after 5 thermal cycles to 1650° F.). (898.9°, the microstructure has clearly changed to include a significant amount of δ -phase at the grain boundaries. This change in microstructure resulting from thermal cycling is reflected in the deterioration in the tensile properties of γ'/γ heat treated and thermally cycled nickel-base superalloy samples presented in Example 3.

[0109] FIG. 21A is a photomicrograph of a surface region of an ATI 718Plus® alloy sample that was δ -phase heat treated as described in Example 1. The thicker white plates primarily on the grain boundaries are δ -phase platelets that result from the δ -phase heat treatment according to non-limiting embodiments of the present disclosure. FIG. 21B is a

photomicrograph of a surface region of the same sample after thermal cycles to a peak sample temperature of 1550° F. (843.3° C.). It may be observed that there is little, if any, difference in the amount of δ -phase platelets after 5 thermal cycles to the 1550° F. (843.3° C.) peak sample temperature. This correlates well with the tensile tests of Example 3, showing that ATI 718Plus® alloy samples δ -phase heat treated as described in Example 1 exhibited a lower variability of tensile properties on thermal cycling.

[0110] FIG. 21C is a photomicrograph of a surface region of an ATI 718Plus® alloy sample that was heat treated according to the conventional γ'/γ heat treatment described in Example 1. It is observed that the microstructure may include a small amount of δ -phase grain boundary precipitates, and that the amount is less than in the samples subjected to the δ -phase heat treatment, as seen in FIG. 21A. However, it is seen in FIG. 21D that after 5 thermal cycles to 1550° F. (843.3° C.), the microstructure has clearly changed to include a significant amount of δ -phase at the grain boundaries. This change in microstructure resulting from thermal cycling is reflected in the deterioration in tensile properties of γ'/γ heat treated and thermally cycled nickel-base superalloy samples presented in Example 3.

[0111] FIG. 22A is a photomicrograph of a surface region of an Alloy 718 sample that was δ -phase heat treated as described in Example 1. The thicker white plates that are primarily on grain boundaries are δ -phase platelets that result from the δ -phase heat treatment according to non-limiting embodiments of the present disclosure.

[0112] FIG. 22B is a photomicrograph of a surface region of the same sample after 5 thermal cycles to a peak sample temperature of 1650° F.). (898.9°. It is observed that there is little, if any, difference in the amount of δ -phase platelets after 5 thermal cycles to 1650° F.). (898.9° peak sample temperature. This correlates well with the tensile tests of Example 3, showing that Alloy 718 samples δ -phase heat treated as described in Example 1 exhibited a lower variability of tensile properties on thermal cycling.

[0113] FIG. 22C is a photomicrograph of a surface region of an Alloy 718 sample that was heat treated according to a conventional γ'/γ heat treatment described in Example 1. It is observed that the microstructure may include a small amount of δ -phase grain boundary precipitates, and that the amount is less than in the samples subjected to the δ -phase heat treatment, as seen in FIG. 22A. However, it is seen in FIG. 22D that after 5 thermal cycles to 1650° F.). (898.9°, the microstructure has clearly changed to include a significant amount of δ -phase at the grain boundaries. This change in microstructure resulting from thermal cycling is reflected in the deterioration of tensile properties of γ'/γ heat treated and thermally cycled nickel-base superalloys presented in Example 3.

[0114] The present disclosure has been written with reference to various exemplary, illustrative, and non-limiting embodiments. It will be recognized by persons having ordinary skill in the art that various substitutions, modifications, or combinations of any of the disclosed embodiments (or portions thereof) may be made without departing from the scope of the invention, as defined solely by the claims. Thus, it is contemplated and understood that the present disclosure embraces additional embodiments not expressly set forth herein. This disclosure is not limited by the description of the various exemplary, illustrative, and non-limiting embodiments, but rather solely by the claims. In this manner, it will be understood that the claims may be amended during pros-

ecution of the present patent application to add features to the claimed invention as variously described herein.

1. A method for heat treating a nickel-base alloy, comprising:

heating a nickel-base alloy to a heat treating temperature in a heat treating temperature range, wherein the nickel-base alloy comprises nickel, chromium, and iron, is strengthened by niobium, and optionally comprises one or more of aluminum and titanium alloying additions;

holding the nickel-base alloy within the heat treating temperature range for a heat treating time sufficient to form an equilibrium or near-equilibrium concentration of δ -phase precipitates in grain boundaries of the alloy and up to 25 percent by weight of total γ' -phase and γ'' -phase within the alloy; and

cooling the nickel-base alloy.

2. The method of claim 1, wherein the heat treating temperature range is in the range of a temperature that is 20° F. (11° C.) greater than the nose of the TTT diagram for delta phase precipitation to a temperature that is 100° F. (55.6° C.) below the nose of the TTT diagram.

3. The method of claim 1, wherein the heat treating time is in a range of 30 minutes to 300 minutes.

4. The method of claim 1, wherein cooling the nickel-base alloy comprises air cooling.

5. The method of claim 1, wherein cooling the nickel-base alloy comprises cooling the alloy at a cooling rate no greater than about 1° F. per minute (0.56° C. per minute).

6. (canceled)

7. The method of claim 1, wherein the nickel-base alloy comprises, in percent by weight: up to 0.1 carbon; 12 to 20 chromium; up to 4 molybdenum; up to 6 tungsten; 5 to 12 cobalt; 6 to 14 iron; 4 to 8 niobium; 0.6 to 2.6 aluminum; 0.4 to 1.4 titanium; 0.003 to 0.03 phosphorus; 0.003 to 0.015 boron; nickel; and incidental impurities; wherein a sum of the weight percent of molybdenum and the weight percent of tungsten is at least 2 and not more than 8; a sum of atomic percent aluminum and atomic percent titanium is from 2 to 6; a ratio of atomic percent aluminum to atomic percent titanium is at least 1.5; and the sum of atomic percent aluminum and atomic percent titanium divided by atomic percent niobium is from 0.8 to 1.3.

8. The method of claim 1, wherein the nickel-base alloy comprises, in percent by weight: 0 to about 0.08 carbon; 0 to about 0.35 manganese; about 0.003 to about 0.03 phosphorus; 0 to about 0.015 sulfur; 0 to about 0.35 silicon; about 17 to about 21 chromium; about 50 to about 55 nickel; about 2.8 to about 3.3 molybdenum; about 4.7 percent to about 5.5 niobium; 0 to about 1 cobalt; about 0.003 to about 0.015 boron; 0 to about 0.3 copper; 12 to 20 iron; aluminum; titanium; and incidental impurities; wherein the sum of atomic percent aluminum and atomic percent titanium is from about 2 to about 6 percent; the ratio of atomic percent aluminum to atomic percent titanium is at least about 1.5; and the sum of atomic percent of aluminum plus titanium divided by atomic percent of niobium equals from about 0.8 to about 1.3.

9. The method of claim 1, wherein the nickel-base alloy comprises, in percent by weight: 0.01 to 0.05 carbon; up to 0.35 manganese; up to 0.035 silicon; 0.004 to 0.020 phosphorus; up to 0.025 sulfur; 17.00 to 21.00 chromium; 2.50 to 3.10 molybdenum; 5.20 to 5.80 niobium; 0.50 to 1.00 titanium; 1.20 to 1.70 aluminum; 8.00 to 10.00 cobalt; 8.00 to 10.00 iron; 0.008 to 1.40 tungsten; 0.003 to 0.008 boron; nickel; and incidental impurities.

10. The method of claim 1, wherein the nickel-base alloy comprises, in percent by weight: 50.0 to 55.0 nickel; 17 to 21.0 chromium; up to 0.08 carbon; up to 0.35 manganese; up to 0.35 silicon; 2.8 to 3.3 molybdenum; greater than 0 to 5.5 niobium and tantalum, wherein the sum of niobium and tantalum is from 4.75 to 5.5; 0.65 to 1.15 titanium; 0.20 to 0.8 aluminum; up to 0.006 boron; iron; and incidental impurities.

11. The method of claim 1, wherein the nickel-base alloy comprises at least one of a foil, a honeycomb core, and a honeycomb panel.

12. The method of claim 1, further comprising, subsequent to cooling the nickel-base alloy, stabilizing heat treating the nickel-base alloy, wherein stabilizing heat treating comprises:

heating the nickel-base alloy to a stabilizing heat treating temperature of 1700° F. (926.7° C.) or less, wherein the heat treating temperature is equivalent to an expected maximum in-service temperature of an article comprising the nickel-base alloy; and

cooling the nickel-base alloy from the stabilizing heat treating temperature.

13. The method of claim 12, wherein cooling the nickel-base alloy from the stabilizing heat treating temperature comprises air cooling.

14. The method of claim 12, wherein cooling the nickel-base alloy from the stabilizing heat treating temperature comprises cooling at a cooling rate no greater than about 1° F. per minute (0.56° C. per minute).

15. A method of heat treating a nickel-base alloy, comprising:

heating a nickel-base alloy to a heat treating temperature in a range of 1700° F. (926.7° C.) to 1725° F. (940.6° C.);

holding the nickel-base alloy at the heat treating temperature for a heat treating time in a range of 30 minutes to 300 minutes; and

air cooling the nickel-base alloy;

wherein the nickel-base alloy comprises, in percent by weight, 17.00 to 21.00 chromium, 2.50 to 3.10 molybdenum, 5.20 to 5.80 niobium, 0.50 to 1.00 titanium, 1.20 to 1.70 aluminum, 8.00 to 10.00 cobalt, 8.00 to 10.00 iron, 0.008 to 1.40 tungsten, 0.003 to 0.008 boron, 0.01 to 0.05 carbon, up to 0.35 manganese, up to 0.035 silicon, 0.004 to 0.020 phosphorus, up to 0.025 sulfur, nickel, and incidental impurities.

16. The method of claim 15, further comprising, after cooling the nickel-base alloy, stabilizing heat treating the nickel-base alloy, wherein stabilizing heat treating comprises:

heating the nickel-base alloy to a stabilizing heat treating temperature that is an expected maximum in-service temperature of an article comprising the nickel-base alloy and is about 1700° F. (926.7° C.) or less; and

air cooling the nickel-base alloy.

17. The method of claim 15, wherein the nickel-base alloy comprises at least one of a foil, a honeycomb core, and a honeycomb panel.

18. A 718-type-nickel-base alloy comprising:

an austenite matrix including grain boundaries;

an equilibrium or near-equilibrium concentration of δ -phase precipitates at the grain boundaries; and

up to 25 percent by weight of total γ' -phase and γ'' -phase precipitates; and

wherein the 718-type nickel-base alloy comprises nickel, chromium, and iron, and is strengthened by niobium, and optionally one or more of aluminum and titanium alloying additions.

19. The 718-type nickel-base alloy of claim **18**, comprising in percent by weight, up to 0.1 carbon, 12 to 20 chromium, up to 4 molybdenum, up to 6 tungsten, 5 to 12 cobalt, 6 up to 14 iron, 4 to 8 niobium, 0.6 to 2.6 aluminum, 0.4 to 1.4 titanium, 0.003 to 0.03 phosphorus, 0.003 to 0.015 boron, nickel, and incidental impurities;

wherein a sum of the weight percent of molybdenum and the weight percent of tungsten is at least 2 and not more than 8;

wherein a sum of atomic percent aluminum and atomic percent titanium is from 2 to 6;

wherein a ratio of atomic percent aluminum to atomic percent titanium is at least 1.5; and

wherein a sum of atomic percent aluminum and atomic percent titanium divided by atomic percent niobium is from 0.8 to 1.3.

20. The 718-type nickel-base alloy of claim **18**, comprising, in percent by weight: 0 to about 0.08 carbon, 0 to about 0.35 manganese; about 0.003 to about 0.03 phosphorous; 0 to about 0.015 sulfur; 0 to about 0.35 silicon; about 17 to about 21 chromium; about 50 to about 55 nickel; about 2.8 up to about 3.3 molybdenum; about 4.7 to about 5.5 niobium; 0 to about 1 cobalt; about 0.003 to about 0.015 boron; 0 to about 0.3 copper; 12 to 20 iron; aluminum; titanium; and incidental impurities; wherein the sum of atomic percent aluminum and atomic percent titanium is from about 2 to about 6 percent; the ratio of atomic percent aluminum to atomic percent titanium

is at least about 1.5; and the sum of atomic percent of aluminum plus titanium divided by atomic percent of niobium equals from about 0.8 to about 1.3.

21. The 718-type nickel-base alloy of claim **18**, comprising, in percent by weight: 0.01 to 0.05 carbon; up to 0.35 manganese; up to 0.035 silicon; 0.004 to 0.020 phosphorus; up to 0.025 sulfur; 17.00 to 21.00 chromium; 2.50 to 3.10 molybdenum; 5.20 to 5.80 niobium; 0.50 to 1.00 titanium; 1.20 to 1.70 aluminum; 8.00 to 10.00 cobalt; 8.00 to 10.00 iron; 0.008 to 1.40 tungsten; 0.003 to 0.008 boron; nickel; and incidental impurities.

22. The 718-type nickel-base alloy of claim **18**, comprising, in percent by weight: 50.0 to 55.0 nickel; 17 to 21.0 chromium; up to 0.08 carbon; up to 0.35 manganese; up to 0.35 silicon; 2.8 to 3.3 molybdenum; greater than 0 to 5.5 niobium and tantalum, wherein the sum of niobium and tantalum is from 4.75 to 5.5; 0.65 to 1.15 titanium; 0.20 to 0.8 aluminum; up to 0.006 boron; iron; and incidental impurities.

23. An article of manufacture made by a process comprising the method of claim **1**.

24. The article of manufacture of claim **23**, wherein the article of manufacture comprises at least one of a face sheet, a honeycomb core, and a honeycomb panel of a thermal protection system for a hypersonic flight vehicle or a space vehicle.

25. An article of manufacture comprising an alloy according to claim **12**.

26. The article of manufacture of claim **25** comprising one of a face sheet, a honeycomb core, and a honeycomb panel of a thermal protection system for a hypersonic flight vehicle.

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