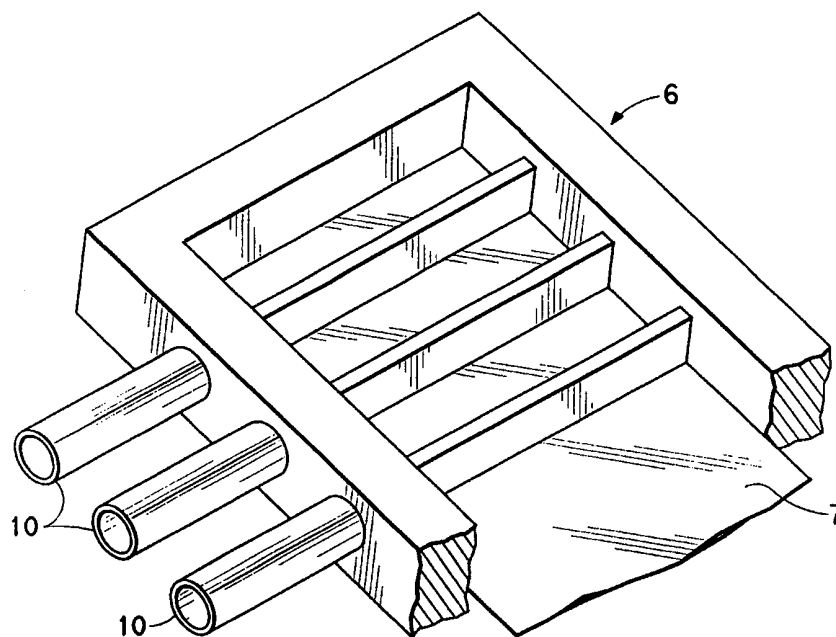




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(54) Title: WINDOW FOILS FOR ELECTRON BEAM PROCESSORS



(57) Abstract

A window foil for use in an electron beam processor comprising a heat-treatable titanium alloy that provides excellent cold formability and increased yield strength as well as high creep resistance at elevated temperatures. The improved creep resistance at high temperatures assures prolonged window foil service. In particular, alpha-beta titaniums and beta titaniums (alloys) that are heat-treatable are of interest to this invention.

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TITLE

WINDOW FOILS FOR ELECTRON BEAM PROCESSORS

Field Of The Invention

5 This invention relates to a novel window foil comprising a titanium alloy exhibiting excellent cold rollability, as well as a combination of excellent oxidation resistance and improved mechanical properties at high temperatures, for use in electron beam processors.

Background Of the Invention

10 In an electron beam processor (EBP), electrons are generated and accelerated in a vacuum then emitted from a chamber to irradiate an object with electrons. The electron beam processor is used in various applications including crosslinking and grafting of polymeric materials, curing of coatings and inks,
15 pasteurization of foodstuff, sterilization of medical products, etc. and so on, wherein the electrons pass through a thin window foil to reach the product treatment area.

 An electron beam processor typically comprises: a) a power supply, b) an electron emitter, c) an accelerator for shaping the emitted particles into a beam and
20 for directing and accelerating the energized particle beam toward an irradiation window, d) an irradiation window comprising a metal foil, e) a window support structure which provides mounting and cooling for the window foil, and f) a vacuum chamber from which air molecules are removed so air cannot interfere with the generation of the particle beam. Electron beam energy is expressed by
25 the acceleration voltage, which is typically in the range of 100 kV to 10,000 kV. The difference between the pressure on the inside of the window foil and the outside is typically 1 atm, and can be as high as 10 atms or more in some applications.

 The window foil in an EBP is typically made of a low atomic number
30 material to minimize the absorption of electrons. The foil must be vacuum-tight and capable of withstanding the pressure differentials in the system. In order to

achieve optimum efficiency, the foil should be very thin. Additionally, the foil should be capable of withstanding high temperatures of 600°F or more.

Window foils in crosslinking applications are often made from pure titanium ("Ti" ASTM Grade 1, 2, or 3) or alpha / near-alpha titanium alloys (ASTM Grade 9). These windows have excellent electron permeability due to the low atomic number of Ti. However, there are a few problems with the prior art windows. Titanium is chemically active at elevated temperatures and will oxidize in air, resulting in the formation of a scale. Hence, the foil undergoes severe corrosion damage by reaction with the atmosphere or special gas outside of the chamber. Ti and its alloys have the tendency to creep at elevated temperatures. (*Creep* (Met.): a time-dependent plastic deformation of metals under steady load, which leads to eventual rupture of the metals). The window foil is subject to relatively high stresses and deformed by the pressure differential between the chamber and the product treatment area. The thin foil is heated by the thermions generated during the passage of the electron beams, raising the foil temperature up to 400°F or more. The stress created by a combination of the pressure differential and the high temperature causes the foil to rupture after a short period of time.

The principal and predominant emphasis in EBP technology development has been in the design and application of the electron beam processor, or in the window support structure and cooling methods. Relatively little attention has been given to alternatives to the conventional window foils which are made out of either titanium or common alpha / near-alpha titanium alloys. Titanium ASTM grade 1, grade 2 and alpha titanium alloy ASTM grade 9 are considered the industry standards. U.S. Patent No. 5,561,342 describes an improved design for an electron beam exit window with a novel supporting grid. The window foil is made from either titanium or titanium alloy (3%Al and 2.5% V, same as alpha titanium alloy ASTM grade 9), or a composite foil of two layers. U.S. Patent No. 5,501,600 describes an irradiator for irradiating a strand with the window foil also from titanium alloy ASTM grade 9. U.S. Patent No. 5,486,703 relates to an electron beam window with improved cooling design, employing a window foil of pure titanium or an alpha titanium alloy similar to a grade 9 (3%Al and 1% V). U.S. Patent No. 4,362,965 discloses an improved support configuration for the

window and evaluates various materials for window foils. The window is a complex foil composite comprising: a) a polyester film and b) a low Z metal film laminated with polyester or vapor deposited on the polyester film, with Z referring to atomic number.

5 A polyethylene crosslinking application typically runs at high energy level with a voltage of 150 to 500kV and a current level between 0.4 and 1.5 Ampere, and an elevated foil temperature of 400°F or more. To reduce energy usage while still allowing an accelerated crosslinking effect, a very thin window foil having a thickness of .005" or less is often used to allow effective electron beam
10 penetration. The thin window foil routinely fails after a short period of time in service due to creep fatigue and corrosion. The foil failure requires excessive downtime to remove the old window foil and replace it with a new one.

 There is thus a need for improvements with regard to a thin window foil having excellent corrosion and mechanical properties at high temperatures, as well
15 as an extended service life as relative to the pure titanium or alpha titanium alloy window foils in the prior art.

Summary of the Invention

 The present invention, according to one aspect, relates to method of
20 constructing a window foil for use with electron beam processors. The invention resides in the discovery that an alpha-beta or essentially beta titanium alloy with inherent good formability and rollability characteristics, after being rolled to a desired thickness to be used as a window foil in an electron beam processor, then subject to heat treatment, develops significantly improved strength and creep
25 resistance at elevated foil temperatures above 400°F, and prolongs the service life of the window considerably over that of a window foil in the prior art.

 The present invention also provides an electron beam processor and the like which generates an electron beam, and a window foil for use with the electron beam processor. The foil comprises a titanium alloy having inherent good
30 formability and rollability characteristics, heat treated to provide improved strength and creep resistance at elevated foil temperatures above 400°F and prolong the service life of the foil.

There is also an electron beam processor and the like which generates an electron beam, and a window foil for use with the electron beam processor. The foil comprises a titanium alloy having inherent good formability and rollability characteristics, rolled to a thickness of 0.005" or less, and heat treated to provide a window foil with excellent electron penetration, a prolonged service life, as well as improved strength and creep resistance characteristics at elevated foil temperatures above 400°F.

Specifically, there is provided a window foil for use with electron beam processors, comprising a foil exhibiting significantly improved strength and creep resistance characteristics, and a significantly longer service life than that of a foil constructed from pure titanium and alpha titanium alloys of the prior art. In a preferred form, the window foil is made from a titanium alloy with inherent good oxidation resistance, good formability and rollability characteristics, consisting essentially of, in weight percent, molybdenum 14 to 16, niobium 2.5 to 3.5, silicon 0.15 to 0.25, aluminum 2.5 to 3.5, and oxygen 0.12 to 0.16, and balance titanium and incidental impurities. The window foil is conventionally formulated and rolled into sheet stock. The sheet stock is then formed to produce a thin foil suitable for electron beam processors. Subsequent heat treatments provide a window foil with surprisingly improved strength and creep resistance characteristics at high temperatures.

Another aspect of the invention is a method of cross-linking polymer materials by electron irradiating, wherein the electron beam irradiator employs a window foil made from a non-alpha titanium alloy having good formability and rollability characteristics, being subject to heat treatment before or after being formed to a desired thickness to develop improved strength and creep resistance characteristics at least twice that of alpha titanium alloys at elevated foil temperatures above 400°F, thus prolonging the service life of the window foil.

A better understanding of the invention will be obtained from the following detailed description and the accompanying drawings and applications of the invention, wherein the features, properties, and relation of elements are described and exemplified.

Brief Description of the Drawings

Figure 1 is a diagram of an embodiment of an electron beam processor.

Figures 2A and 2B are perspective views showing various embodiments of a window support structure.

5 Figure 3 is a graph showing the temperature distribution profile in a typical window foil during the irradiation process.

Figure 4 is a graph showing the tensile properties of the preferred titanium alloy used in the window foil of the present invention as a function of temperature.

10 Figure 5 is a graph showing the creep properties of the preferred titanium alloy used in the window foil of the present invention as a function of temperature and time under plastic deformation.

Description of the Preferred Embodiments

15 Figure 1 shows an embodiment of an electron beam processor (EBP) or irradiator according to this invention. As known in the art, an EBP includes a housing which provides an enclosure defining a vacuum chamber 1. The interior of a chamber 1 is maintained under vacuum conditions by a vacuum pump 2. In operation, electron beam is emitted from an electron generating source 3, which
20 consists of an energized filament made of metal such as tungsten and heated by a power source. The electrons 9 that are generated by this heating are accelerated by an electron accelerating means 20. This electron accelerating means 20 consists of a cathode 4 and anode 5. The electrons are accelerated by the electric field created by high voltage that is applied to cathode 4 and anode 5. The
25 electrons 9 pass out of the chamber through an electron beam permeable window foil 7 and strike the product 8 to be irradiated.

Figures 2A and 2B show a perspective view of various embodiments of the window foil 7 and its window supporting frame 6. The typical material of construction for the support structure is copper or beryllium copper, designed to
30 remove heat from the window foil 7. In Fig. 2A, pipes 10 are used to cool the support structure and the window foil 7 with a coolant such as pressurized nitrogen. Other cooling methods may be employed in conjunction with and/or

separate from the cooling pipes such as the use of fins or slots between the pipes to increase the heat removal. Figure 2B shows another embodiment of a window, with the window supporting frame 6 being a perforated water-cooled support structure to cool the window foil 7 with water-cooled pipes. Besides those shown
5 above, other window support structures and cooling methods well known in the art may also be employed as well.

Cooling of the window structure is necessary to remove the continuous heat build-up in the window foil in an irradiation process. As the electron beams pass through the foil, a portion of the energy is absorbed in the foil, raising the foil
10 temperature. Figure 3 is a profile showing the local axi-symmetric temperature distribution in a window foil during the irradiation process, from the center of the window foil at a temperature of up to 950°F (510°C) to the edge of the window foil adjacent to the window frame at about 400°F (200°C). The thermal profile analysis was done using a computer model of the operation of an electron beam
15 processor. In this computer model, a vacuum load is first applied to a window foil of 0.0006" thick supported by a copper window frame with counter-flow cooling water at 25°C to remove heat from the support frame, then the foil is heated in an irradiation process.

A window foil as employed in an electron beam processor as described
20 and modeled above is widely employed in various industrial applications, such as polymerization of materials. In these applications, electrons are directed toward the surface of a polymeric material, such as a film, and causing chemically reactive species such as free radicals or ions to form. The free radicals are capable of undergoing or participating in a host of secondary reactions such as cross-
25 linking of the polymer, degradation of the polymer, recombination reactions and grafting co-polymerization or polymerization.

Since the variability of properties within titanium and alloys, the basis for the material used in the present invention, depends to some degree on the specific chemistry and thermal-mechanical history of the material, it is desirable to present
30 a limited explanation of the nature of titanium and its alloys. Titanium has two elemental crystal structures, one is a closed-packed hexagonal (commonly known as alpha), the other a body-centered cubic (commonly known as beta). The beta

structure is found only at high temperatures (above the beta transus temperature or also known as the alpha / beta processing temperature), unless the titanium is alloyed to maintain a beta structure at lower temperatures. The addition of other alloying elements to a titanium base will favor either the alpha or beta forms.

5 As might be expected, there is a range of titanium and alloy compositions, some of which tend to embody the characteristics of alpha and others which are more similar to beta. The window foils in the prior art employ pure titaniums ASTM grades 1 and 2, an alpha rich alloy ASTM grade 9 and variations thereof. Pure titaniums are known for their excellent corrosion characteristics. Alpha rich
10 titanium alloys generally exhibit excellent creep strength characteristics. However, pure titaniums and alpha-structured alloys as used in window foils of the prior art cannot be heat-treated to develop higher mechanical properties because they are single-phased. Furthermore, they generally have poor malleability or fabricability to be formed into thin window foils. Malleability or
15 fabricability characteristic is typically expressed as cold reduction rate. Cold reduction is the change in thickness from the original to the final state before annealing is required. Cold reduction of alpha rich titanium alloys typically ranges from about 25-30%. Cold reduction rate for alpha-beta titaniums or beta alloys is typically in the range of about 35% to 50%.

20 Titaniums with a mixture of alpha and beta phases -- alpha-beta titaniums, and beta titanium alloys or essentially beta titanium alloys that are heat-treatable are of interest to this invention. These alloys lend themselves to improvement for a window foil application through microstructure tailoring. The microstructure is produced by heat treatment for about over ½ hour at a temperature below the beta
25 transus temperature to impart the titanium alloy with higher strength, ductility, and resistance to fatigue crack initiation. When the alloys undergo this type of heat treatment and upon cooling from this temperature, they develop a microstructure consisting of fine, equiaxed regions of alpha grains interspersed and bonded with the thin films of beta in the alloys.

30 Until now, titanium alloys with a mixture of alpha and beta phases (alpha-beta titaniums), beta titanium alloys or essentially beta titanium alloys that are heat-treatable have not been attempted or disclosed as suitable for window foils in

electron beam applications. Applicants have found that when a thin foil made from the titanium alloys of the type under consideration undergoes heat treatments through microstructure tailoring, higher mechanical properties which are particularly suitable for window foil applications are obtained.

5 1. Suitable alpha-beta titanium alloys include:

 Ti-5Al-2Sn-2Zr-4Mo-4Cr with excellent creep resistance properties, known as Ti-17;

 Ti-6Al-2Sn-4Zr-6Mo with short-term strength as well as long-term creep strength, also known as Ti-6246;

10 Ti-7Al-4Mo with improved creep resistance at temperatures up to 900°F, known as UNS:R56740;

 Ti-4.5Al-3V-2Mo-2Fe is hardenable with superior superplastic formability properties, also known as SP-700;

15 Ti-4Al-3Mo-1V with good formability and a good combination of strength and temperature stability up to 900°F, known as Ti-431;

 Ti-5Al-5Sn-2Zr-2Mo-0.25Si offers good tensile, stress rupture, and creep resistance properties in high temperature applications of 800 to 1000°F, known as TI-5522-S; and

20 Ti-8Mn with excellent formability and intermediate strength for temperature up to 600°F; known as UNS:R56080.

 2. Beta and essentially beta alloys contemplated for use according to the invention, but not limited to, include the following:

 Ti-11.5Mo-6Zr-4.5Sn with excellent cold formability and strength potential, known as Beta III;

25 Ti-3Al-8V-6Cr-4Mo-4Zr - known as UNS:R58640, excellent for rolling into very thin foil;

 Ti-10V-2Fe-3Al for high-strength and toughness applications at temperatures up to 600°F that can be further strengthened through heat treatments, known as Ti-10-2-3;

30 Ti-13V-11Cr-3Al with excellent strengths; known as UNS:R58010

 Ti-15V-3Al-3Cr-3Sn, known and readily available as Ti-15-3, with good workability and creep resistance and thermal stability after heat treatments;

Ti-8Mo-8V-2Fe-3Al with good formability and age hardening characteristics, known as Ti-8823;

Ti-15Mo-3Al-2.7Nb-0.25Si with improved oxidation resistance, creep resistance, and thermal stability, known as Ti-21S; and

5 Ti-15Mo-5Zr for excellent cold-formability

Although it would be impossible for Applicants to test every possible titanium alloy listed above, once an ordinary artisan reads this specification and the accompanying claims, and thereby learns of the advantages of heat-treating an alpha-beta, beta, or essentially beta titanium alloy having inherent excellent
 10 fabricability characteristics to develop higher mechanical properties in the alloy at elevated temperatures, such an artisan will be able to employ the titanium alloy in window foil applications.

A preferred titanium alloy for reasons of operability, commercial availability, and economics is disclosed in U.S. Patent No. 4,980,127 to Titanium
 15 Metals Corporation of America ("Timet"). Commercial products which incorporate the titanium alloy disclosed in this patent are sold by Timet of 400 Rouser Road, Pittsburgh, PA 15230, under the trademark Timetal® 21S. Timet® 21S, or Ti-15Mo-3Al-2.7Nb-0.25Si, possesses inherent excellent
 20 improved mechanical properties and thermal stability after heat treatments. Timet® 21S has a nominal composition comprising 15 percent molybdenum, 3 percent aluminum, 3 percent niobium or columbium, and 0.2 percent silicon, with a beta transus temperature about 1485°F or 807°C. In its most preferred form, the alloy has the following composition in weight percent:

- | | | |
|----|--------------------------|-------------------|
| 25 | 1. molybdenum: | 14.0 to 16.0; |
| | 2. niobium or columbium: | 2.4 to 3.2; |
| | 3. aluminum: | 2.5 to 3.5; |
| | 4. silicon: | 0.15 to 0.25; and |
| | 5. iron: | up to 0.4. |

30 Variations of the above alloy are also available with added palladium or with no aluminum at all.

Prior to processing in accordance with the present invention, the manufacturer of the alloy will formulate an alloy in accordance with the nominal composition, keeping in mind the ranges listed above. The alloy is usually cast into an ingot at relatively high temperatures. The ingot is then forged to a rolling bar or slab having a thickness of about 4 to 5 inches or so. The rolling bar stock is heated to a temperature generally on the order of about 1750°F to about 1950°F then run through a rolling mill to be rolled to a coil. The cold rolling and annealing is repeated until a sheet of desired thickness is achieved. The material may be surface-conditioned by pickling, grinding, trimming and etching in-between the rolling steps as required. The material is then shipped as coils of strips in gages between 0.012" to 0.1" thick. The material is typically provided in the beta solution-treated condition, which precipitates α to provide strengthening on aging.

Thereafter, in accordance with the present invention, the thickness of the solution-treated coils is preferably further reduced by either hot or cold forming to the desired thickness for a window foil. For purposes of the present invention and to take full advantage of the excellent fabricability of the alloy, the foil should preferably be rolled to the ultimate window thickness in the range of 0.0004" to .005". A thick foil prolongs service life at the expense of energy consumption. Thin window foils maximize electron penetration and lower energy usage at the expense of shortened life.

If the strip cannot be cold-rolled, a hot process on a hand-mill using cover sheets to form packs for heat retention is the other viable option. Although the pack process offers the opportunity to cross-roll to minimize texture, it is labor-intensive and inherently a lower-yield process. To obtain maximum mechanical strength improvement, a duplex annealing treatment may be employed. The treatment begins with a solution annealing at a temperature about 50 to 100°F below the beta transus temperature of the alloy. Additionally, exposure of solution heat-treated material to temperatures of 500-800°F should be kept to less than one hour to avoid the possibility of embrittlement. If forming temperature exceeds the beta transus temperature (about 1485°F for the preferred Ti alloy Ti-

15Mo-3Al-2.7Nb-0.25Si), time at such temperature should be minimized to avoid excessive grain growth. Therefore, cold forming is most preferred.

In a preferred cold forming process, the material is then annealed at a temperature below the beta transus temperature, on the order of 1450°F or less, and run through a multi-stand rolling mill. The cold rolling and annealing is repeated until a sheet of the desired thickness is achieved. The cold rolling process may incorporate intermediate vacuum or inert gas annealing. In this step, if the coil is not received from the manufacturer in a solution-treated condition, then the foil is first solution annealed before being cooled in a vacuum tower at a relatively rapid pace.

After final forming, additional annealing treatments such as single or duplex aging treatments are imposed on the foil to achieve improved mechanical properties. In the preferred embodiment, the additional aging is done after the foil is rolled to the desired thickness. However, it should be noted that the heat treatments can be done prior to final thickness with the only expense being the loss of some ductility and thus it is more difficult to roll the foil to the final desired thickness in the downstream process.

In the additional heat treatment process, aging causes decomposition of the super-saturated beta phase retained on quenching, or transformation of the martensite to $\alpha + \beta$ phase. In this process, the titanium foil is preferably wrapped or rolled around a hollow mandrel with a large diameter, forming a roll. The foil should be loosely wrapped around the mandrel and forming a roll having a thickness of 1/4" or so in foil, so as to allow heat penetration to all layers of the roll for an even and uniform aging. The roll is then aged in a vacuum oven or inert gas oven kept at a temperature on the order of about 800°F to about 1300°F, and heated at such a rate that the titanium roll reaches the aging temperature in the absolute minimum amount of time.

It is preferred that the window foil of the desired thickness be single-aged at temperatures on the order of about 800°F to about 1300°F for over 1/2 hour, preferably for a period ranging from 2 to 16 hours, and most preferably on the order of 8 hours. It is most preferred that the aging should be at 1150°F for 7 hours, then at a reduced temperature of 1100°F for another hour. Aging over 60

hours may provide higher strength, but will decrease ductility and fracture toughness.

After aging, the titanium roll should be cooled to room temperature at a preferred cooling rate of about 200°F per hour.

5 For electron beam processors operating at elevated temperatures up to about 1200°F, a duplex age is recommended. The window foil is initially aged at 800°F to about 1300°F for a period over ½ hour as in the single-aged process described above, then followed by another aging at about 800°F to about 1200°F for at least another ½ hour (“duplex aging”). The duplex age is recommended to
10 retain ductility. In the first aging stage, the high temperature “weakens” the grains relative to the grain boundaries, and the second aging stage stabilizes the grains against embrittlement.

It is very important, in fact critical, that the foil be aged for electron beam irradiation applications at temperatures above 400°F to improve mechanical
15 properties of the titanium alloy, and thus the window foil. If the foil is not aged, potential exists for embrittlement in the foil by the precipitation of omega phase or very fine alpha. It is also critical that care be taken during aging to avoid heating too slowly, because this can result in very high strength with concomitant low ductility. Additionally, temperature controls with an upper cutoff be employed to
20 prevent temperature from exceeding beta transus if solution-treated titanium coils are used as the starting materials.

As with all titaniums and alloys, the material can be stress relieved without adversely affecting its strength or ductility. Stress relieving treatments decrease undesirable stresses that may result from cold forming or thermal stresses from the
25 heat treatment process. During stress relief, care should be taken to prevent overaging to lower strength. This includes cooling from stress relief temperature to about 900°F at a preferred rate of less than 100°F per hour. Below this temperature, cooling is optional. Oil or water quenching is not recommended since this can induce residual stresses.

30 Vacuum or inert gas furnaces of the electrical resistance type are most preferable for aging the material. Temperature control equipment should be accurate to $\pm 2^\circ\text{F}$ and controllable to within $\pm 15^\circ\text{F}$.

Aging results in a window foil with significantly improved mechanical properties. Ultimate tensile strength of the resulting titanium alloy is on the order of 150-180 Ksi, and ranging upward as high as 210Ksi with ductility still above 2%. Figure 4 serves to demonstrate the tensile strength properties over a wide
5 range of temperatures up to 1100°F (600°C) of the preferred titanium alloy, Ti-15Mo-3Al-2.7Nb-0.25Si, used in the window foil of the present invention after strip aging at 1100°F for 8 hours.

Table 1 is a compilation of data comparing and contrasting the room temperature properties of prior art materials, pure titanium ASTM grades 1, 2, and
10 3, and alpha titanium alloy ASTM grade 9, with the preferred titanium alloy of the invention before and after heat treatment at various aging temperatures. It should be noted that the higher the aging temperature, the lower the improved yield strength and tensile strength, and the higher the ductility.

Table 1 - Comparable properties - ultimate strength and yield strength at room T

Material	Room T Ultimate Strength* (Ksi)	0.2% Yield Strength* (Ksi)	% Elong.
Pure Ti Grade 1	35	25	24
Pure Ti Grade 2	50	40	20
Pure Ti Grade 3	65	55	18
Alpha Ti Grade 9	90	70	15
Ti-15Mo-3Al-2.7Nb- .25Si UnAged	128	125	15
Ti-15Mo-3Al-2.7Nb- .25Si aged 1000°F, 8hrs., air cool	191	178	6
Ti-15Mo-3Al-2.7Nb- .25Si Aged 1100°F, 8hrs., air cool	164	151	8
Ti-15Mo-3Al-2.7Nb- .25Si Aged 1275°F, 8hrs., air cool	125	115	10

At elevated temperatures over 600°F, which is the area of interest for the present invention, there is a marked difference between the mechanical properties of the prior art materials and the titanium alloys of the present invention as shown in Table 2 below:

Table 2 - Yield strength at elevated temperatures

Material	Yield strength (Ksi) at 797°F	Yield strength (Ksi) at 1004°F
Pure Ti Grade 3	17	13
Ti-15Mo-3Al-2.7Nb-.25Si Aged 1000°F, 8hrs., air cool	142	90
Ti-15Mo-3Al-2.7Nb-.25Si Aged 1100°F, 8hrs., air cool	142	90

The improvement in creep resistance strength over the prior art material is remarkable as shown in Table 3 below for the preferred alloy, Ti-15Mo-3Al-2.7Nb-0.25Si, after strip aging.

Table 3 - Creep strength at elevated temperatures at 0.5% strain.

Material	Creep Strength (Ksi) 1000 hrs. at 698°F	Creep Strength (Ksi) 1000 hrs. at 797°F	Creep Strength (Ksi) 1000 hrs. at 1004°F
Pure Ti Grade 3	-	<4	1
Ti-15Mo-3Al-2.7Nb-.25Si Aged 1000°F, 8hrs., air cool	87	46	11
Ti-15Mo-3Al-2.7Nb-.25Si Aged 1100°F, 8hrs., air cool	87	46	11

Figure 5 is a plot that shows the creep results over a wide temperature range for the preferred alloy Ti-15Mo-3Al-2.7Nb-0.25Si, after strip aging at 1100°F for 8 hours. In this figure, "creep" is shown as a time-dependent plastic deformation of metals under steady load. The x axis is a function of the temperature (°C) as well as time under load in hour ('t'): $P = (°C + 273) (20 + \log t)/1000$.

It is expected that after being heat-treated, a window foil employing the preferred titanium alloy, Ti-15Mo-3Al-2.7Nb-0.25Si, retains the inherent

excellent oxidation resistance properties relative to pure titaniums exhibited by unaged Ti alloys even at high temperatures as shown in Table 4 below:

Table 4 - Oxidation resistance at various test temperatures

Material	Test T °F	<u>Weight gain mg/cm²</u>			
		24 hrs.	48 hrs.	72 hrs.	96 hrs.
Pure Ti Grade2	1200	0.50	0.72	1.00	1.11
	1500	7.30	14.35	20/64	26.10
Ti-15Mo-3Al-2.7Nb- .25Si UnAged	1200	0.14	0.23	0.27	0.32
	1500	1.21	1.75	2.06	2.88

5 The following examples are intended to illustrate to one of ordinary skill as how to make and use the present invention. They are not intended to limit the scope of the Letters Patent granted hereon.

EXAMPLE 1

10 A titanium alloy is formulated having a nominal composition of 15 percent molybdenum, 3 percent aluminum, 3 percent niobium or columbium, and 0.2 percent silicon. The balance is titanium with the trace elements being maintained below the maximum level set forth in the preferred composition above. The material is forged, rolled and surfaced conditioned into a sheet of .05" thick and rolled into a 50 lb. coil.

15 A first specimen is cut from the sheet and cold-rolled to a foil of 0.0005" thick and appropriate width covering the electron beam window size, rolled into a coil about 500 feet long. A most preferable range of .0005" to .0009" provides an optimum thickness that allows for maximum electron penetration, low energy consumption, as well as a window life at least 5 times that of the prior art under
20 similar conditions.

 The specimen is aged at 1150°F for 7 hours, then 1100°F for 1 hour, and then air-cooled. The specimen has an ultimate yield strength of about 140 Ksi at an elevated temperature of 800°F. A sheet of foil appropriate size covering the window frame is cut from the coil and tautly extended over a window frame.

Thereafter, the window foil is subject to operation in accordance with the procedures of an electron beam processor.

After an aging treatment as described above, the window employs the preferred titanium alloy is expected to have a service life extended in proportion with the improvement in strength and creep resistance property as compared with the pure titanium alloys and the alpha titanium alloys of the prior art under the same operating conditions.

EXAMPLE 2

A titanium alloy with a nominal composition of 15 percent molybdenum, 3 percent aluminum, 3 percent niobium or columbium, and 0.2 percent silicon, is processed as above forming a sheet of .05" thick and rolled into a 50 lb. coil.

A sheet is cut from the coil, and cold-rolled into a coil with an ultimate thickness of 0.0005". The foil is then duplex-aged, first aging at 1275°F for 8 hours, then duplex aging at about 1200°F for another 8 hrs. A sheet of foil of the appropriate size covering a window frame is cut from the coil and tautly extended over a window frame. The duplex aging gives the foil the prerequisite maximum long-term thermal stability for elevated temperature operations up to 1200°F.

It should be noted that after suitable duplex aging heat treatment as described above, the window employs the preferred titanium alloy is metallurgically stable for at least 1000 hrs. at a temperature up to 1140°F.

The present invention has been described broadly and in relation to its preferred embodiments. One of ordinary skill will be able to effect various changes, substitutions of equivalents, and other alterations without departing from the broad concepts disclosed herein.

What is claimed is:

1. A window foil for use with an electron beam processor and the like which generates an electron beam, comprising:
 - 5 a) a frame connected to the electron beam processor;
 - b) a vacuum-tight metal foil of a thickness which is permeable to the electron beam;
 - c) a supporting structure for said metal foil for mounting on said frame;
- 10 wherein said metal foil comprises a heat-treatable titanium alloy exhibiting improved yield strength characteristics of at least 50% over that of commercially pure titaniums of same thickness under similar conditions at room temperature, and good cold formability and cold rollability to permit at least about 35% cold reduction.
- 15
2. The high-power window foil of claim 1, wherein said titanium alloy is heat-treatable at a temperature of about 800°F to about 1300°F for at least about ½ hour to develop improved yield strength characteristics at least twice that of commercially pure titaniums of same thickness under similar conditions at
20 elevated foil temperatures of at least 400°F.
3. The window foil of claim 1, wherein said titanium alloy has good oxidation resistance exhibited by a weight gain of about one half of commercially pure titaniums of same thickness under similar time at similar temperature
25 conditions of elevated foil temperatures of at least 400°F.
4. The high-power window foil of claim 1, wherein said titanium alloy exhibits a creep resistance property of at least twice that of commercially pure titaniums of same thickness under similar conditions of elevated foil temperatures
30 of at least 400°F.

5. The high-power window foil of claim 1, wherein said titanium alloy contains at least one alloying element from the group consisting of: molybdenum, niobium, silicon, aluminum, and oxygen.
- 5 6. The high-power window foil of claim 1, wherein said titanium alloy consists essentially of, in weight percent, molybdenum 14 to 16, niobium 2.5 to 3.5, silicon 0.15 to 0.25, aluminum 2.5 to 3.5, and oxygen 0.12 to 0.16, and balance titanium and incidental impurities.
- 10 7. An electron beam processor comprising:
- a) a housing defining a vacuum chamber;
 - b) means for generating a particle beam within the vacuum chamber;
 - c) means for accelerating the particle beam consisting of a cathode and an anode;
 - 15 d) means for directing the particle beam;
 - e) a vacuum-tight metal foil which is permeable to the electron beam; and
 - f) a supporting structure for said metal foil for mounting on said frame;
- 20 wherein:
- said metal foil comprises a heat-treatable titanium alloy exhibiting good cold formability and cold rollability to permit at least about 35% cold reduction,
 - said heat-treatable titanium alloy exhibits improved yield strength
 - 25 characteristics of at least 50% over that of commercially pure titaniums under similar conditions at room temperature.
8. The electron beam processor of claim 7, wherein said titanium alloy consists essentially of, in weight percent, molybdenum 14 to 16, niobium 2.5 to
- 30 3.5, silicon 0.15 to 0.25, aluminum 2.5 to 3.5, and oxygen 0.12 to 0.16, and balance titanium and incidental impurities.

9. A process for fabricating window foils suitable for an electron beam processor operating at a high energy level and for high temperature applications, from a rolled strip of a titanium alloy consisting essentially of, in weight percent, molybdenum 14 to 16, niobium 2.5 to 3.5, silicon 0.15 to 0.25, aluminum 2.5 to 3.5, and oxygen 0.12 to 0.16, and balance titanium and incidental impurities, said method comprising the steps of:

- a) cold rolling said rolled strip to foil of a thickness between 0.0004" to .009";
- b) aging said cold-rolled strip of foil at a temperature of from about 800 to about 1300°F so as to develop improved yield strength characteristics of at least twice times that of commercially pure titaniums under similar conditions of elevated foil temperatures of at least about 400°F.

10. An improved method for irradiating an object with the use of an electron beam processor with the use of an electron beam processor that comprises: a) a housing defining a vacuum chamber, b) means for generating a particle beam within the vacuum chamber, c) means for accelerating the particle beam consisting of a cathode and an anode, d) means for directing the particle beam, e) a vacuum-tight metal foil which is permeable to the electron beam; and f) a window supporting structure for said foil for mounting on said frame,

said improved method comprises:

- a) generating and propagating said particle beam toward said metal foil;
- b) irradiating said object with said particle beam;

wherein:

said metal foil comprises a titanium alloy heat-treatable for improved yield strength characteristics of at least twice that of commercially pure titaniums under similar conditions of elevated foil temperatures above 400°F, and good cold formability and cold rollability to permit at least about 35% cold reduction.

30

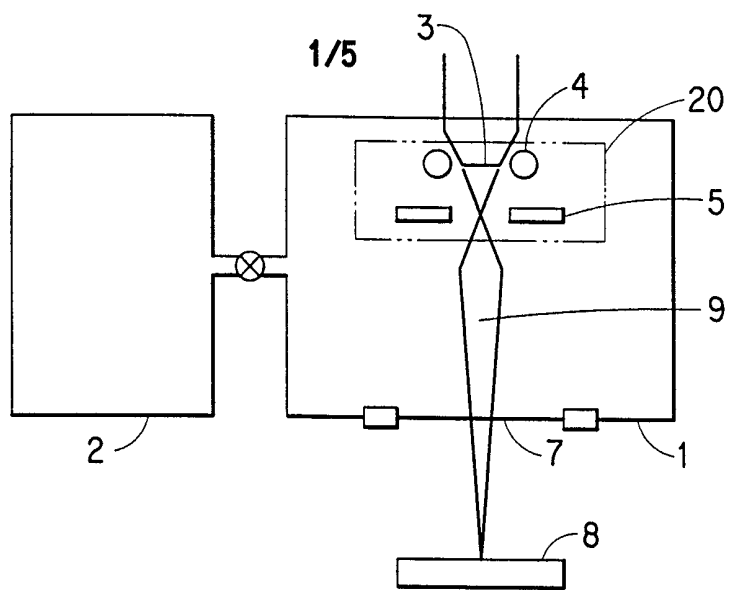


FIG. 1

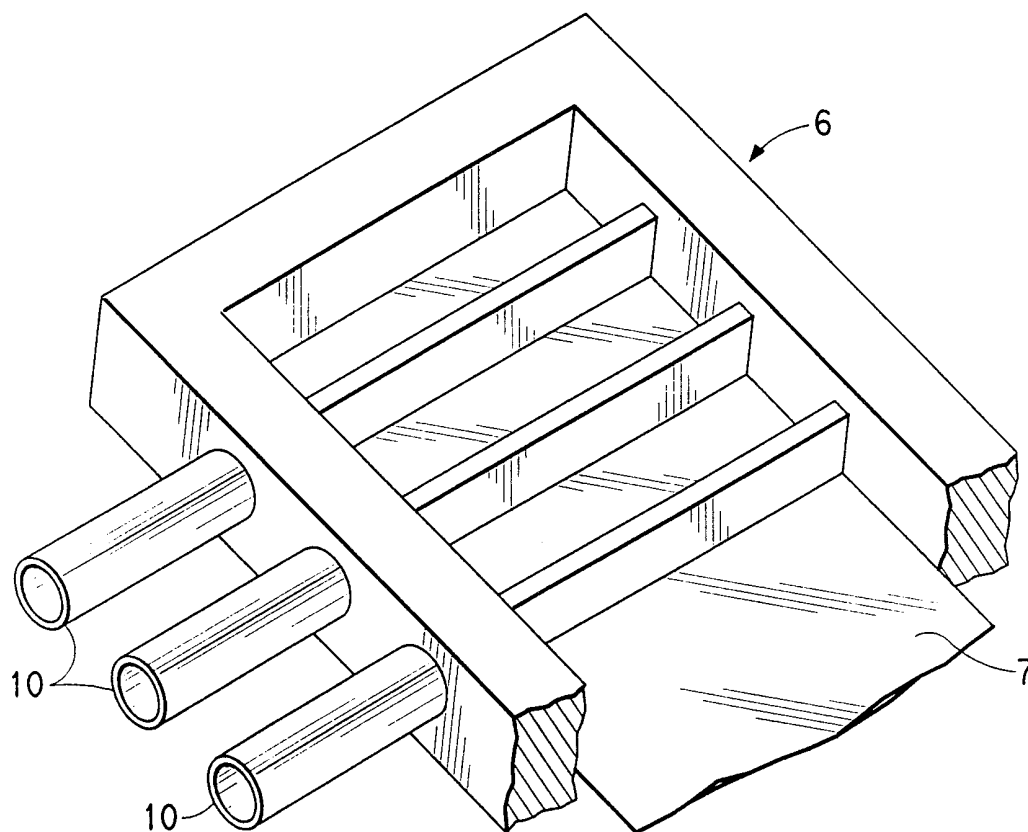


FIG. 2A

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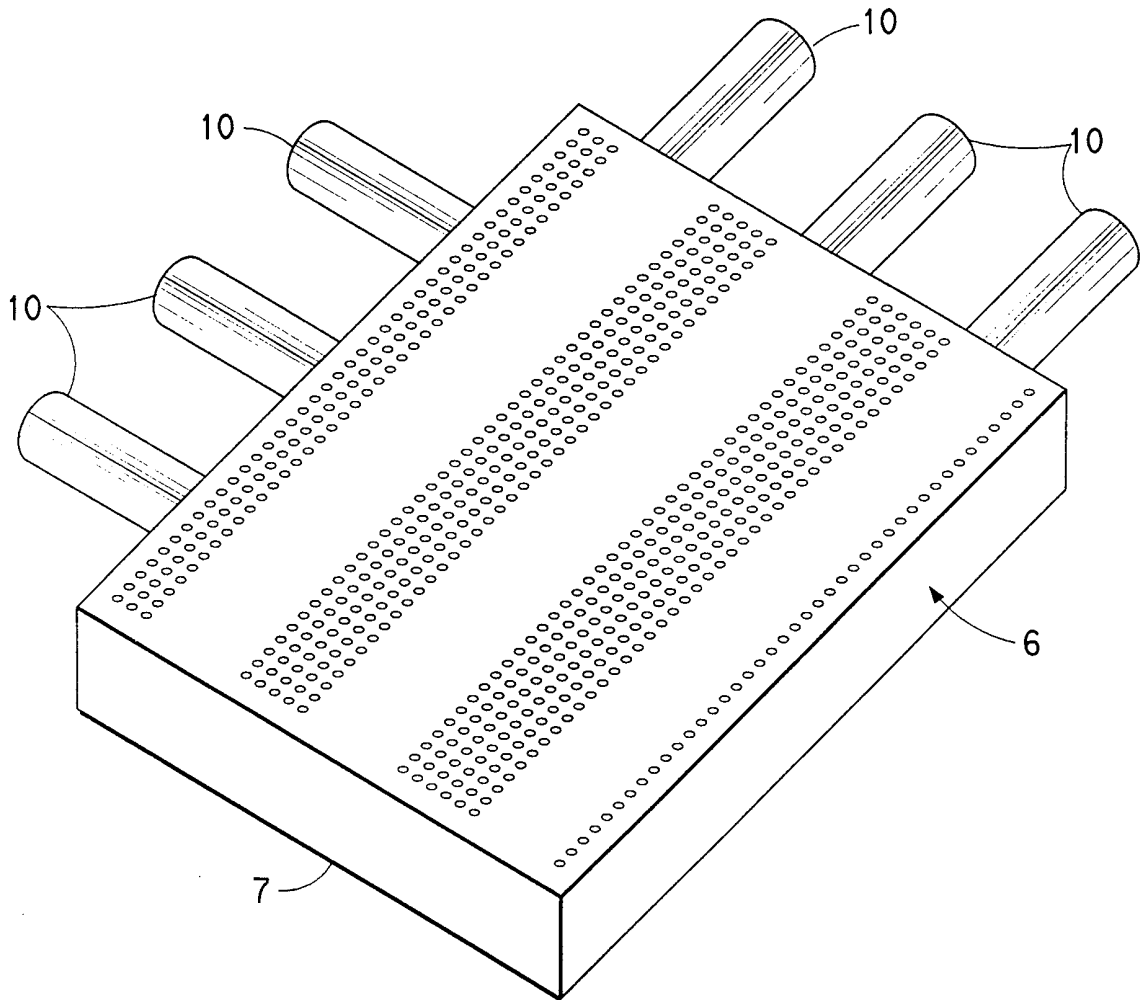


FIG. 2B

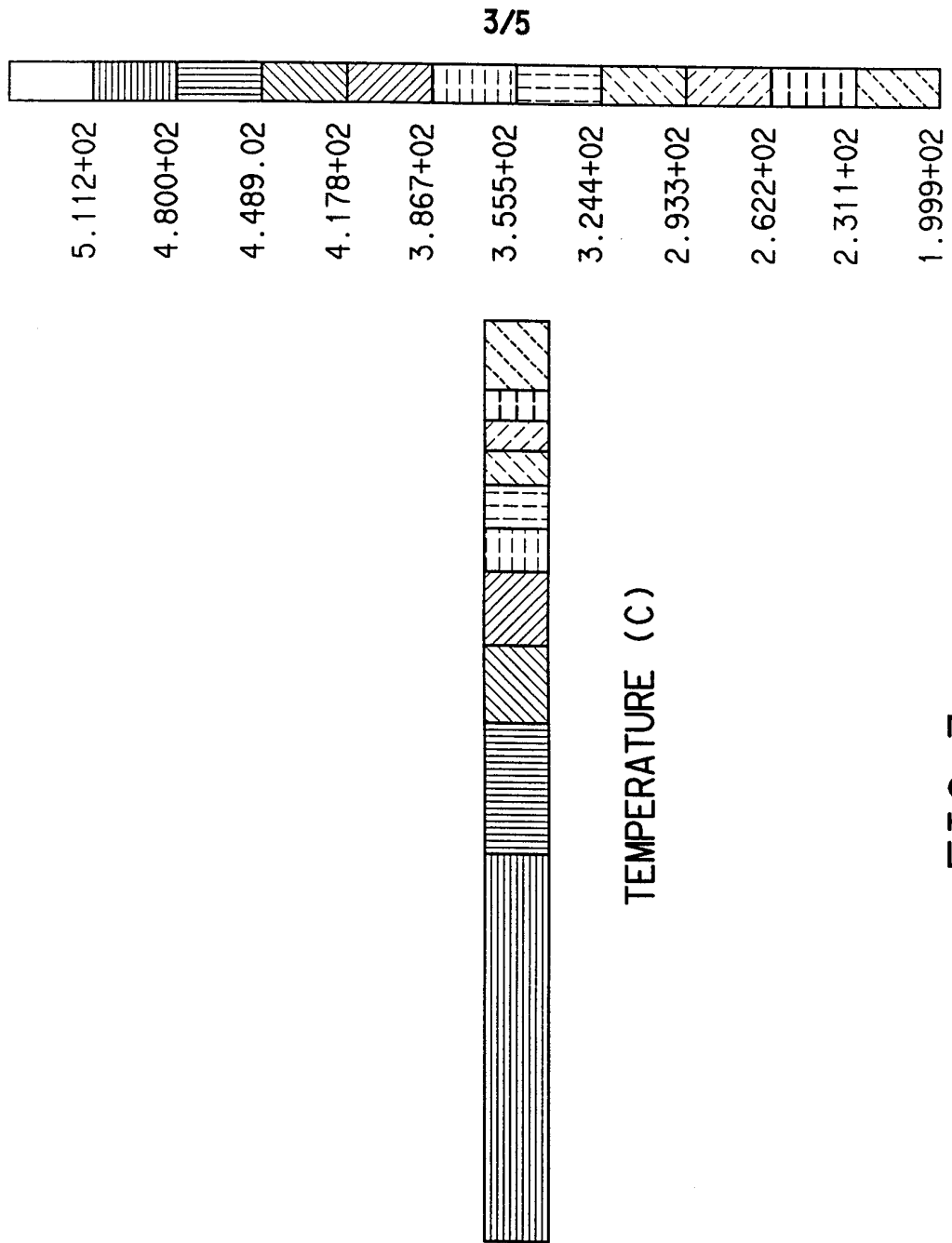


FIG. 3

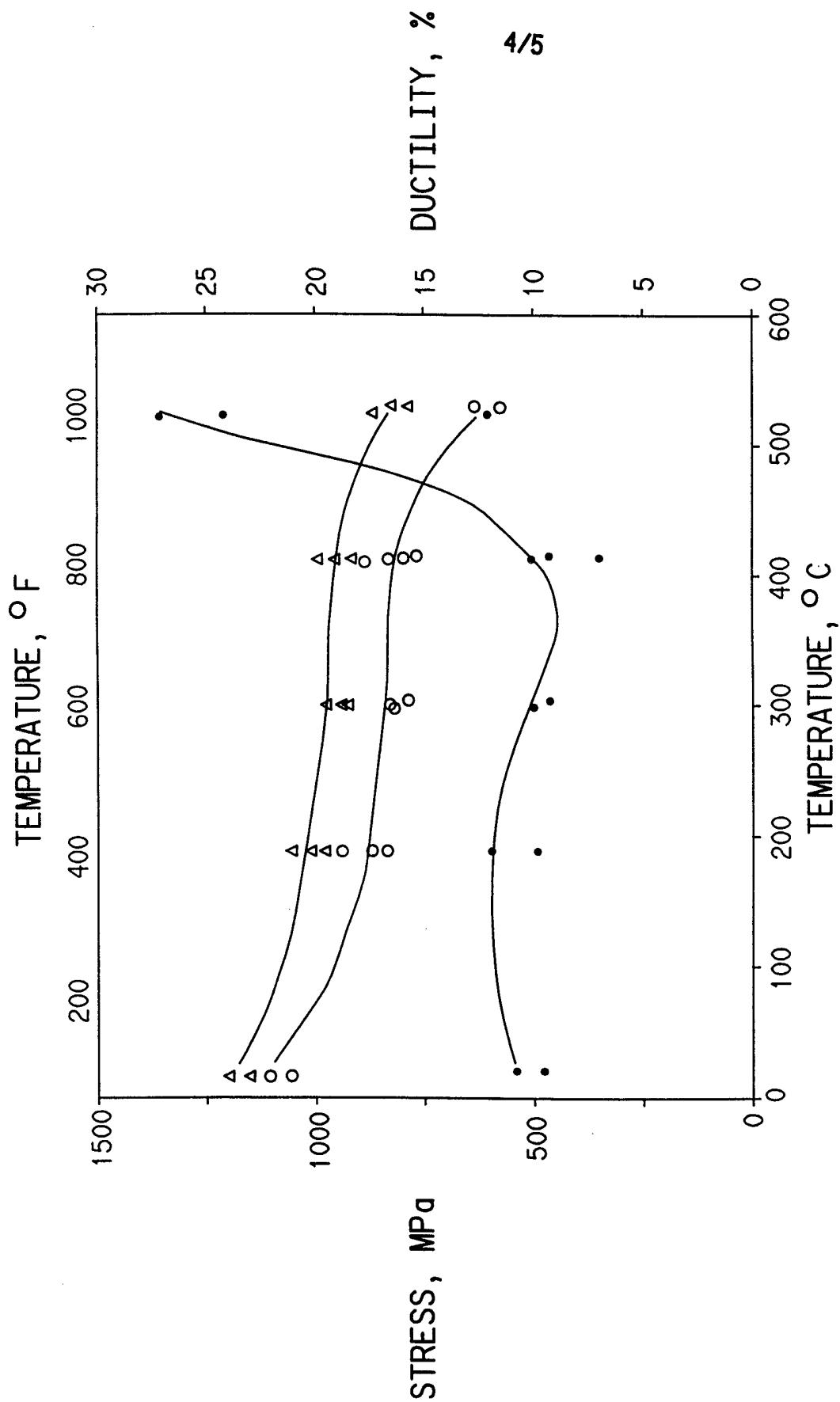


FIG. 4

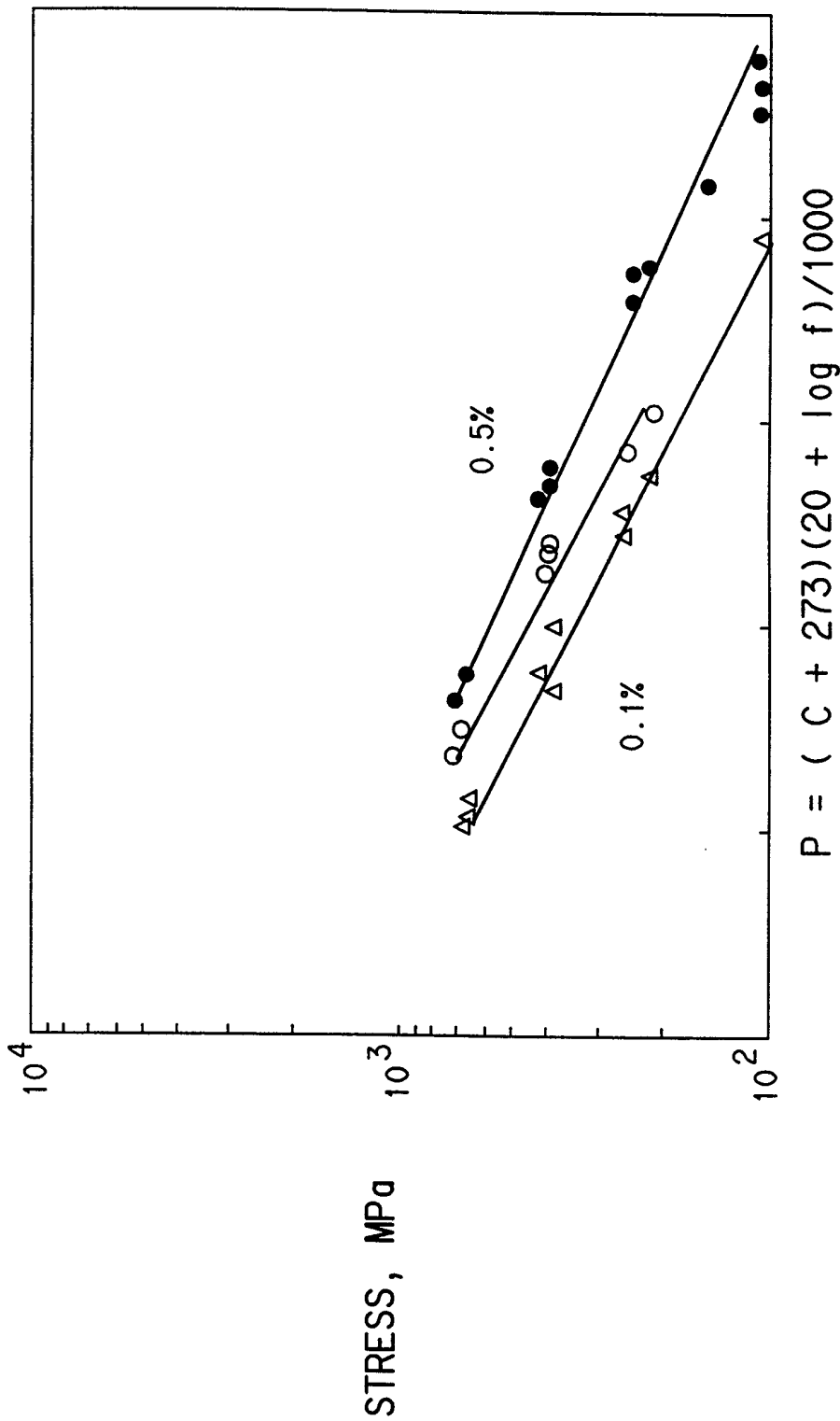


FIG. 5

INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 99/00233

A. CLASSIFICATION OF SUBJECT MATTER
 IPC 6 H01J33/04 H01J5/18

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 H01J C22C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5 486 703 A (LOVIN JOSEPH R ET AL) 23 January 1996 cited in the application see column 1, line 27 - line 32	1,7,10
Y	US 4 980 127 A (PARRIS WARREN M ET AL) 25 December 1990 cited in the application see abstract see column 1, line 23 - line 37	1,7,10

Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
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- "P" document published prior to the international filing date but later than the priority date claimed

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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Date of the actual completion of the international search

19 April 1999

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

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