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ANNEALING TITANIUM WIRE

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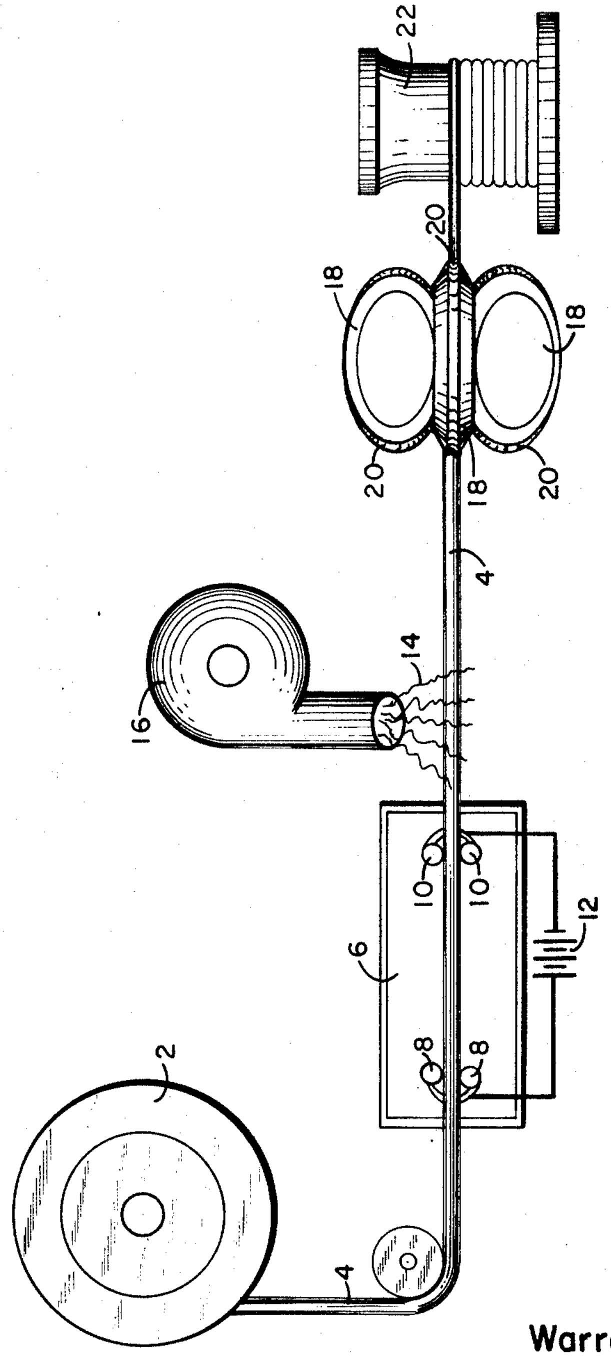

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ANNEALING TITANIUM WIRE

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ABSTRACT OF THE DISCLOSURE

Method of reducing the cross section of titanium and titanium base alloy wire which has been previously hot rolled at a temperature below its beta transes comprising heating the wire to a temperature between the beta transes and about 100° F. above the beta transes followed by cooling to below the beta transes and cold drawing.

This invention relates to heat-treating wire and more particularly to annealing titanium or titanium base alloy wire to provide improved cold-rolling characteristics.

Conventional wire processing involves first hot-rolling to form a relatively large diameter wire which is subsequently cold drawn, often with several passes, through a die to provide the desired finished diameter. Instead of drawing through a die, the wire may be cold worked to finished diameter by passing through multiple circumferential rollers having grooved edges. Adjacent edges of the working rollers form a circular aperture so that the wire is worked down to provide the desired finished diameter by a rolling instead of a simple drawing operation.

A serious problem arises in production of such cold formed titanium and titanium alloy wire. Initial hot-rolling, most often accomplished at a temperature below the beta transes temperature and in the alpha-beta or alpha temperature field, produces a definite texture or preferred crystal orientation in the hot-rolled wire. When wire of this type of texture or orientation is subsequently cold-rolled through a multiple roller finishing machine, the resistance of the wire to deformation is not uniform and the cross section of the cold-rolled wire will not be uniform and may actually be oval.

Summarized briefly, the process of this invention comprises heat-treating titanium and titanium alloy wire at a temperature above its beta transes to eliminate the crystal orientation imparted by previous hot-rolling at a temperature below its beta transes. This particular heating step, I have discovered, particularly in the case of titanium alloys, must be carried out so that the wire is heated rapidly to the heat-treating temperature, and then preferably cooled rapidly to prevent the formation of large-size beta grain structure. When the wire is heat-treated in this manner, being quickly raised to temperature and then quickly cooled, it can be reduced to final gauge through a roller type wire drawing machine to produce wire of circularity substantially improved over similar wire drawn without such heat-treatment. In addition, the ductility of the wire will be high so that its cross sectional area may be reduced by a substantial percentage in a single pass through the roller wheels without cracking or breaking of the wire.

The process of this invention may be more readily understood by reference to the single figure of the drawings in which a drum or coil of wire, which has previously been hot-rolled at a temperature below its beta transes, is shown at **2**. The wire **4** is directed into resistance heating furnace **6** in which are arranged two sets of contact rollers indicated at **8** and **10** respectively, these two sets of rollers being spaced apart along the wire and each connected to a pole of a suitable source of electric

current **12**. The section of wire **4** between contact rollers **8** and **10** will complete the electrical circuit, and because of its own internal resistance, will be very rapidly raised to high temperature. Immediately on emergence from furnace **6** the wire is exposed to a blast of cooling air **14** provided by blower **16**.

After cooling, the wire is pulled through a set of edge-grooved rolls **18**, these in the illustration being a set of three arranged around the wire at a radial angle of 120° each to the other with their edge grooves **20** forming a circular aperture through which the wire **4** is pulled by action of bull block **22** actuated by a suitable motor (not shown).

For simplicity and clarity and to illustrate the essential steps of the process, only one set of rolls **18** have been shown in the drawing. In practice, at least a pair of such sets, preferably nested to provide close coupling, will generally be employed so that the wire may be reduced in diameter in a plurality of steps. The first set of a pair may advantageously reduce the wire and give it a curved-side triangle cross section or oval or other non-circular shape, with the second and final set of rolls providing a circular aperture to impart the desired circular cross section.

The wire whose cross section is to be reduced by the method of this invention is characterized by having been previously worked at a temperature below its beta transes. It is common practice in wire manufacture to work commercially pure and alpha and alpha-beta type titanium base alloys in the alpha-beta field or alpha field because this provides easiest plastic deformation without raising the temperature of the material to a point where rapid oxidation and surface deterioration takes place. Moreover, working in the beta field will generally result in larger grains which are difficult to break down in subsequent working at lower temperatures. The process of this invention is to be applied to wire whose latest prior working will have been hot-rolling at a temperature below its beta transes because this type of rolling apparently produces a detrimental crystal structure which affects subsequent cold-rolling when the wire is drawn through edge-grooved rolls. Apparently hot-rolling at such temperatures results in an orientation of the metal crystals so that subsequent deformation will not be uniform in all directions. Particularly when the hot-rolled wire is drawn through edge-grooved rolls the ease of deformation in a plane perpendicular to the length of the wire will not be uniform at various radial angles in this plane. Consequently, such wire when cold drawn through edge-grooved rolls will deform more readily in certain areas or directions, and this will often result in an oval wire instead of having desired circular cross section. It appears that the edge-grooved rolls, no matter how strongly built and precisely aligned to produce a circular orifice through which the wire is drawn, can be deformed elastically to some extent so that wire having preferred crystal orientation, when passing through such rolls, will not produce a truly circular or uniform reduced cross section wire.

According to this invention, however, heating the wire to above its beta transes and subsequently cooling the wire with attention to rapid heating and cooling, for the titanium base alloys particularly, results in an annealed wire of uniform crystal orientation which, when cold drawn through edge-grooved rolls, can be reduced to produce a circular wire of substantially uniform diameter.

The wire should be heated to a temperature definitely above its beta transes temperature, which may be determined for commercially pure titanium or any of the alpha or alpha-beta type titanium base alloys by metallurgical methods well known in the art. The minimum time that the wire is maintained above its beta transes temperature is not critical, since apparently transformation takes place almost instantaneously; and once the wire has been raised to above its beta transes and subsequently cooled, the time above the beta transes temperature, even though but a few seconds, will be sufficient to produce the desired transformation of the crystal structure. It is preferred to heat the wire to a temperature between its beta transes and 100° F. above its beta transes in order to be sure that all portions of the wire have definitely been raised to above the beta transes temperature. Preferably the heat treating temperature is between the beta transes temperature and about 50° F. above the beta transes. While the temperature should be sufficiently higher than the beta transes to insure complete transformation, it is not desirable to raise to excessive temperature since this inevitably results in increased time above the beta transes, and also results in a tendency towards formation of large beta grains.

The time for the heat-treating step of this invention is extremely critical, and for alpha and alpha-beta type titanium base alloys the total elapsed time for heating the wire from room temperature to above its beta transes plus the time it is maintained above its beta transes should not be greater than one minute. Preferably for best results with wire of the recited titanium base alloys the time should not be more than 30 seconds. For commercially pure titanium, the time is not so critical. I have found some improvement with shorter times, but wire of commercially pure titanium may be heat-treated through a cycle from room temperature to above the beta transes plus the time at or above the beta transes, which takes no more than one hour.

The short time requirements for the heat-treating step of this invention requires means for heating wire very rapidly to high temperature. This can readily be accomplished by resistance heating or induction heating. In resistance heating, heavy electrical currents are passed through a limited section of the wire, and the current flow results in extremely rapid heating of the wire. It is no problem to raise the wire temperature to above its beta transes temperature in a matter of seconds when passing a sufficient amount of current. In induction heating, the wire is subjected to high frequency induced electric current, and this also will raise the temperature of the wire extremely rapidly; and raising to above the beta transes by induction heating can be accomplished well within the time limits hereinbefore recited. When heat-treating commercially pure titanium wire, and for which the time is not so critical, coils of wire may be furnace heat-treated, which takes appreciably longer than the other two methods suggested, but total heat-treating time may still be maintained within a desirable period under an hour.

Once the heat-treated wire has been raised to above its beta transes temperature, it is then quickly cooled, at least down through its beta transes. This may be accomplished by any convenient means providing a relatively rapid cooling. Quenching, which involves submerging the hot wire in water or oil and results in an almost instantaneous cooling, can be employed, if desired, although such a fast cooling effect is not essential to satisfactory results according to this invention. It is sufficient that the temperature be rapidly reduced, and to accomplish this, air cooling will produce a relatively fast and efficient reduction of wire temperature from its annealed temperature down to room temperature and particularly at least through the beta transes temperature.

After heat-treatment and subsequent cooling, the wire is cold drawn through a set of edge-grooved rolls to produce circular wire of substantially uniform diameter.

Table 1, following, provides examples of heat-treating commercially pure titanium wire above its beta transes temperature and subsequently cold drawing this through two sets of nested edge grooved rolls to produce wire of improved circularity. The wire was of commercially pure, unalloyed titanium designated in the trade as Ti–55A. The original ingot of the commercially pure titanium was worked down through various stages, the wire being rolled on a hot mill starting at a temperature of about 1750° F. and finishing at a temperature of about 1300° F. The latest prior working (at 1300° F.) was in the alpha field. Table 1 shows the heat-treatment prior to drawing through the edge-grooved rolls, the starting diameter of the wire, which was measured as maximum and minimum diameters along a 10-foot length of wire, and also the diameter measured in the same manner after cold drawing through the edge-grooved rolls.

TABLE 1

Heat-Treatment Prior to Drawing	Starting Diameter[1] (inch)	Variation in Starting Diameter (inch)	Diameter After Drawing [1] (inch)	Variation in Diameter of Cold Drawn Wire (inch)
As Mill Annealed	.266–.277	0.011	.246–.251	0.005
1,300° F. (2 Hrs.) AC [2]	.271–.280	0.009	.246–.257	0.011
Heat-Treated Above Beta Transes:				
1,750° F. (10 Sec.) AC [2]	.264–.274	0.010	.2465–.248	0.0015
1,750° F. (10 Sec.) AC [2]	.267–.276	0.009	.2465–.248	0.0015
1,750° F. (15 Min.) AC [2]	.258–.271	0.013	.246–.247	0.001
1,750° F. (30 Min. AC [2]	.259–.272	0.013	.246–.248	0.002
1,750° F. (1 Hr.) AC [2]	.265–.275	0.010	.246–.248	0.002

[1] Maximum and minimum diameters along 10-foot length of wire.
[2] Total time heating to temperature and air cooling.

It will be seen from Table 1 that with a heat-treatment below the beta transes with subsequent rapid cooling, the variation in wire diameter will be improved from about 0.011 inch to 0.005 inch in the mill annealed condition when heated at 1300° F. When heat-treatment above the beta transes is employed, however, the variation in wire diameter is reduced from a starting figure of between 0.013 inch and 0.009 inch down to a maximum of 0.002 inch, and under the most rapid heating and cooling conditions, less even than this. The beta transes of the Ti–55A commercially pure titanium metal was 1675° F. so that the beta heat-treatments used were all about 75° above the beta transes temperature of the alloy. It will be seen from the variation in diameter of the cold drawn wire that the tests run using longer heating and cooling periods, that is 30 minutes to 1 hour, showed less favorable variation in wire diameter than the tests run at shorter periods of time.

Table 2, following, provides examples of heat-treating alpha-beta type titanium base alloy above its beta transes temperature and subsequently cold drawing this through multiple edge-grooved rolls to produce wire of improved circularity. The wire was of an alloy comprising 8% aluminum, 1% molybdenum, 1% vanadium, and balance substantially all titanium. The original ingot of titanium base alloy was worked down through various stages, the wire being rolled on a hot mill starting at a temperature of about 1950° F. and finishing at a temperature of about 1400° F. to 1500° F. The latest prior working (at 1400° F. to 1500° F.) was in the alpha-beta field. Table 2 shows the heat-treatment prior to drawing through the edge-grooved rolls, the starting diameter of the wire, which was measured as maximum and minimum diameters along a 4-foot length of wire, and also the diameter measured in the same manner after drawing through the edge-grooved rolls. The variations in starting diameters and in the diameters after drawing through the edge-grooved rolls as well as the reduction in area and observations with respect to cracking during drawing are also shown in the table.

TABLE 2.—8Al-1Mo-1V ALLOY

	Starting Diameter [1], (inch)	Variation in Starting Diameter, (inch)	Diameter After Drawing [1], (inch)	Variation in Diameter of Cold Drawn Wire, (inch)	Reduction in Area, Percent	Remarks
Heat-Treatment Prior to Drawing: 1,850° F. (5 min.) AC [2] (α-β Anneal).	.276-.279	0.003	.243-.249	0.006	21.5	Severe Cracking.
	.279-.282	0.004	.254-.260	0.006	15.5	OK, No Cracks.
	.276-.280	0.004	.252-.256	0.004	16.5	Do.
	.276-.278	0.002	.252-.256	0.004	16.0	Do.
Average		0.0035		0.005		
Heat-Treated Above Beta Transes: 1,950° F. (10 Sec.) AC. [2]	.280-.285	0.005	.245-.247	0.002	24	Do.
	.279-.283	0.004	.245-.246	0.001	24	Do.
	.281-.283	0.002	.245-.247	0.002	24	Do.
	.279-.283	0.004	.244-.246	0.002	24	Do.
Average		0.004		0.002		

[1] Maximum and minimum diameters along 4-foot length of wire.
[2] Total time heating to temperature and air cooling.

It will be seen from Table 2 that with a heat-treatment below the beta transes and followed by subsequent rapid cooling, the variation in wire diameter will not be improved in the case of the 8Al-1Mo-1V alloy. The test results indicate that after a heat-treatment at 1850° F. the variation in starting diameter averaged 0.0035 inch; and this was made worse by cold drawing, the average after this step being 0.005 inch. It will also be seen from the results shown in the upper portion of Table 2 that cold drawing to produce a reduction in area of as much as 21.5% resulted in severe cracking of the product wire, although reductions in the range of 15.5% to 16.5% could be obtained with no cracks. When the 8Al-1Mo-1V wire was heat-treated at a temperature above its beta transes, that is at 1950° F. (its beta transes being 1900° F.), and rapidly cooled by air cooling, the variation in diameter before and after drawing through the edge-grooved rolls was reduced from an average of 0.004 inch to an average of 0.002 inch. Thus it is seen that the critical heat-treating and rapid cooling resulted in a substantial improved circularity and uniformity of diameter of the cold drawn wire. Additionally, it will be noted that after heat-treating according to this invention and cold drawing through the edge-grooved rolls, a reduction in area of 24% could be readily obtained without cracking of the wire product, and this compares with substantially less reduction obtainable without the specific heat-treatment employed according to this invention and shown in the upper part of the table.

Table 3, following, provides examples of heat-treating an alpha type titanium base alloy above its beta transes temperature and subsequently cold drawing this through multiple edge-grooved rolls to produce wire of improved circularity. The wire was of an alloy containing 5% aluminum, 2.5% tin, and the balance substantially all titanium.

The original ingot of the titanium base alloy was worked down through various stages, the wire being rolled on a hot mill starting at a temperature of about 1850° F. and finishing at a temperature of about 1300° F. to 1400° F. The latest prior working (at 1300° F. to 1400° F.) was in the alpha field. Table 3 shows the heat-treatment prior to drawing through the edge-grooved rolls, the starting diameter of the wire, which was measured as maximum and minimum diameters along a 4-foot length of wire, and also the diameter measured in the same manner after drawing through the edge-grooved rolls. The variations in starting diameters and in the diameters after drawing through the edge-grooved rolls as well as the reduction in area, and observations with respect to cracking during drawing, are also shown in the table.

TABLE 3.—5Al-2.5Sn ALLOY

	Starting Diameter, [1] (inch)	Variation in Starting Diameter, (inch)	Diameter After Drawing, [1] (inch)	Variation in Diameter of Cold Drawn Wire (inch)	Reduction in Area, Percent	Remarks
Heat-Treatment Prior to Drawing: 1,850° F. (5 Min.) AC [2] (α-β Anneal).	.287-.305	0.018	.261-.268	0.007	20	Severe Cracking.
	.287-.306	0.019	.267-.273	0.006	16.5	OK, No Cracks.
	.288-.308	0.020	.264-.273	0.009	19	Do.
	.290-.306	0.016	.264-.273	0.009	19	Do.
	.286-.306	0.020	.264-.271	0.007	18	Do.
	[3] .264-.271	0.007	[3] .242-.247	0.005	32	Severe Cracking.
Average		0.016		0.007		
Heat-Treated Above Beta Transes: 1,950° F. (10 Sec.) AC [2]	.293-.311	0.018	.261-.264	0.003	24.5	OK, No Cracks.
	.291-.307	0.016	.262-.264	0.002	22.5	Do.
	.289-.303	0.014	.260-.263	0.003	22.0	Do.
	.290-.306	0.016	.260-.263	0.003	22.0	Do.
Average		0.016		0.003		
	[3] .261-.264	0.003	[3] .244-.246	0.002	34	Do.
	[3] .260-.263	0.003	[3] .244-.246	0.002	34	Do.
	[3] .260-.263	0.003	[3] .244-.246	0.002	34	Do.
	[3] .262-.264	0.002	[3] .244-.246	0.002	34	Do.
Average		0.003		0.002		

[1] Maximum and minimum diameters along 4-foot length of wire.
[2] Total time heating to temperature and air cooling.
[3] Second reduction without intermediate anneal.

It will be seen from Table 3 that with a heat-treatment below the beta transes with subsequent rapid cooling, the variation in wire diameter was improved in the case of the 5Al-2.5Sn titanium base alloy only from an average of 0.016 inch to an average of 0.007 inch. In addition, it will be seen that severe cracking occurred when sufficient working was employed to provide a reduction in area during cold drawings through the edge-grooved rolls of 20%. When the reduction was reduced below 20%, cracking was avoided. The last result in the group of tests in which the alloy wire was heat-treated at 1850° F. prior to cold drawing through the edge-grooved rolls was given a second reduction without intermediate anneal so that the total reduction amounted to 32%. Apparently the alloy wire was not sufficiently ductile to maintain its integrity under this degree of reduction and severe cracking resulted.

In the group of tests which were heat-treated above the beta transes of the alloy, namely at 1950° F. and then rapidly reduced in temperature by air cooling, the average variation in starting diameter was reduced from 0.016 inch down to 0.003 inch. The beta transes of the 5Al–2.5Sn alloy is 1900° F. In the upper group of tests in which the wire had been heat-treated above its beta transes, it will be seen that reduction in area of as high as 24.5% could be obtained without any deleterious effect on the wire, that is without formation of cracks. In the lower group of tests in Table 3, also heat-treated at 1950° F. and rapidly reduced in temperature by air cooling, the wire was given a second reduction without intermediate anneal to provide a total reduction in area of 34%. The variation in starting diameter was reduced under these conditions into stages, that is from an original 0.016 inch down to 0.003 inch and then from 0.003 inch down to 0.002 inch in the second reduction. While the improvement in variation of diameter in the cold drawn wire in the second reduction stage is insignificant and also immaterial (since the starting diameter variation was already extremely low) a significant effect is seen in the amount of reduction which can be obtained without cracking in the product wire. It is shown under the test conditions that the wire can be reduced a total of 34% and still produce good wire without cracks. In the case of wire not heat-treated above its beta transes and shown as the bottom test of the upper group in Table 3, a combined reduction of 32% resulted in severe cracking in the wire product.

I claim:

1. A method for reducing the cross section of wire of metal selected from the group consisting of commercially pure titanium and alpha-type and alpha-beta-type titanium base alloys, the latest prior working of said wire being hot-rolling below its beta transes temperature, which comprises:

(a) heating said wire to a temperature between its beta transes temperature and about 50° F. above its beta transes temperature and subsequently cooling said wire to below its beta transes temperature, the total elapsed time for heating said wire from room temperature to above its beta transes temperature plus the time it is maintained above its beta transes temperature being not greater than one minute; and,

(b) cold drawing said wire through edge-grooved rolls, whose edge grooves in adjacent alignment form a circular aperture to provide a circular wire of substantially uniform diameter.

2. A method for reducing the cross section of wire of metal selected from the group consisting of commercially pure titanium and alpha-type and alpha-beta-type titanium base alloys, the latest prior working of said wire being hot-rolling below its beta transes temperature, which comprises:

(a) heating said wire to a temperature between its beta transes temperature and about 100° F. above its beta transes temperature and subsequently cooling said wire to below its beta transes temperature, the total elapsed time for heating said wire from room temperature to above its beta transes temperature plus the time it is maintained above its beta transes temperature being not greater than one minute; and,

(b) cold drawing said wire through edge-grooved rolls whose edge grooves in adjacent alignment form a circular aperture to provide a circular wire of substantially uniform diameter.

3. A method as set forth in claim 2 including passing electric current through said wire after said hot-rolling whereby said heating is accomplished by the resistance of said wire to the passage of said electric current.

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