Jan. 9, 1968 TOSHIMITSU TANABE 3,362,854

SURFACE HARDENING TREATMENT OF STEEL OR ALLOY STEEL BY MEANS OF SUPERSONIC WAVE

Filed Feb. 4, 1964

3 Sheets-Sheet 1

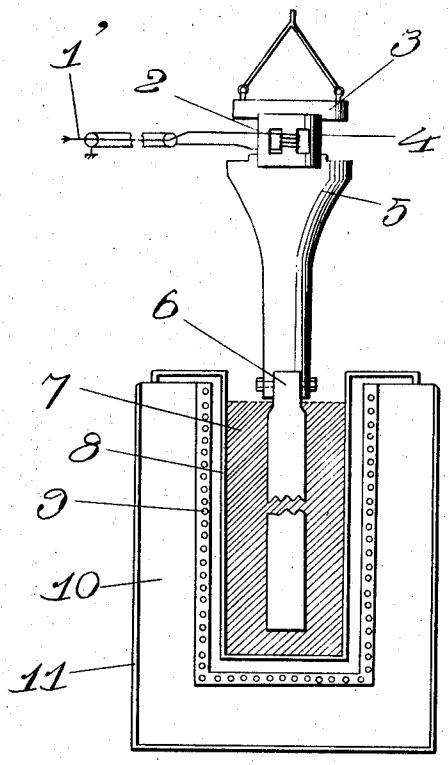
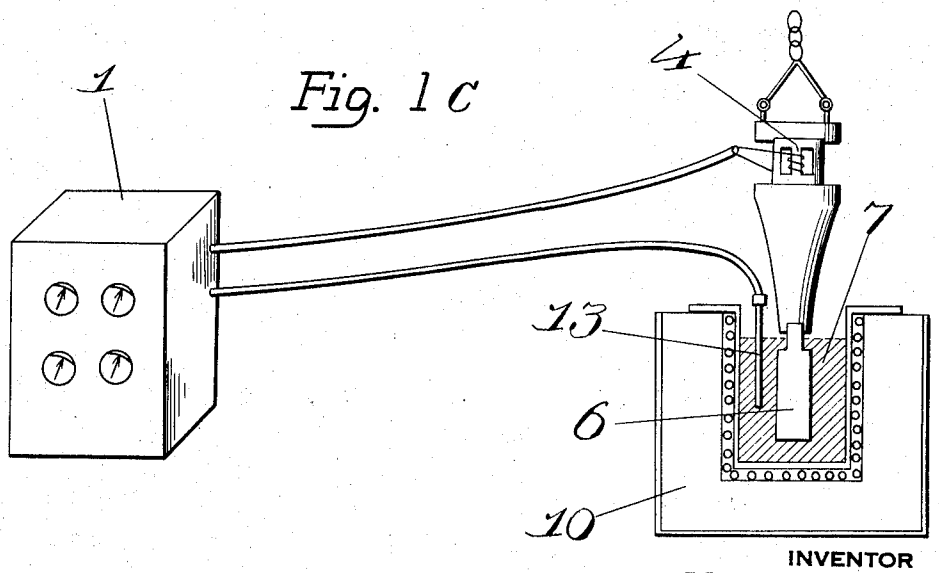


Fig. 1a

Fig. 1c



INVENTOR

Toshimitsu Tanabe

BY Otto John Munn.

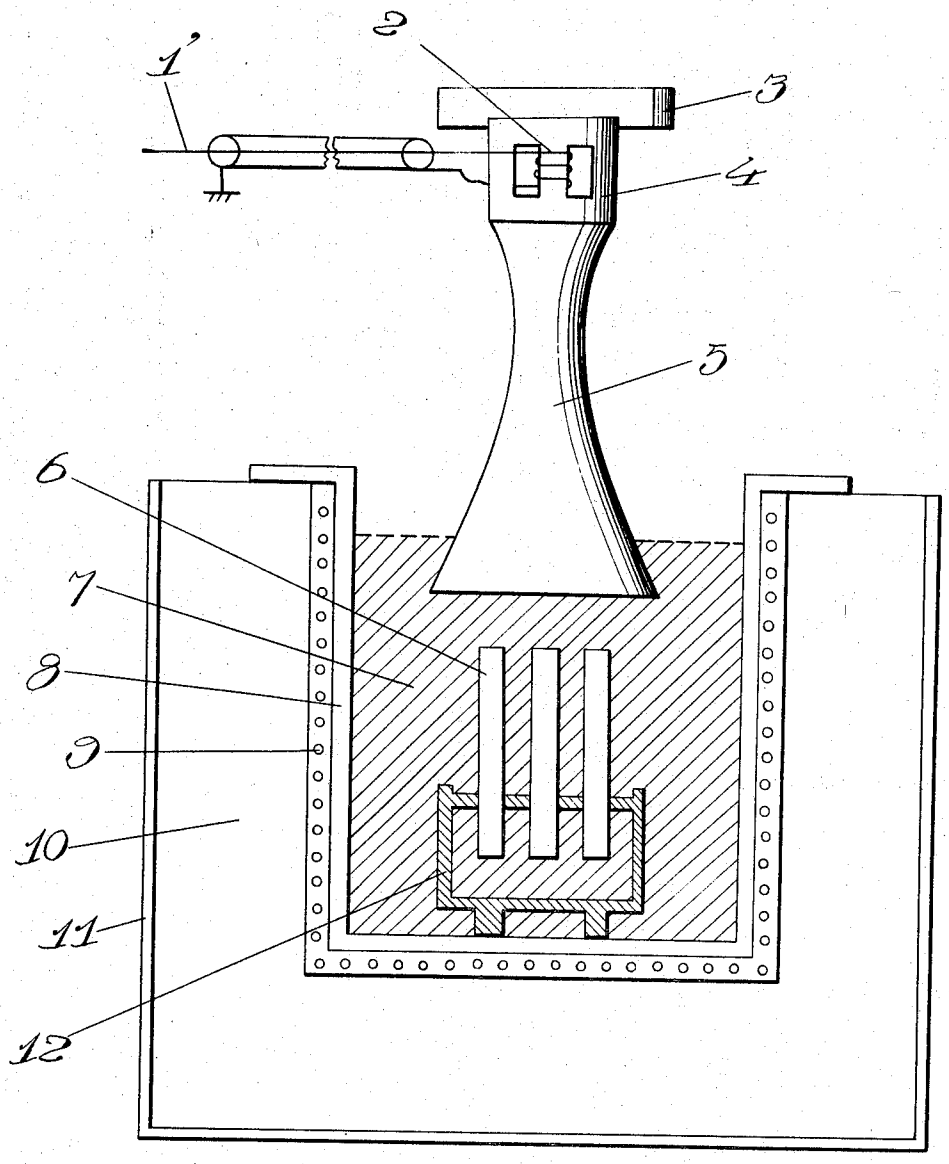
ATTORNEY

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Fig. 1b



INVENTOR
Toshimitsu Tanabe
BY Otto John Munn
ATTORNEY

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Fig. 2

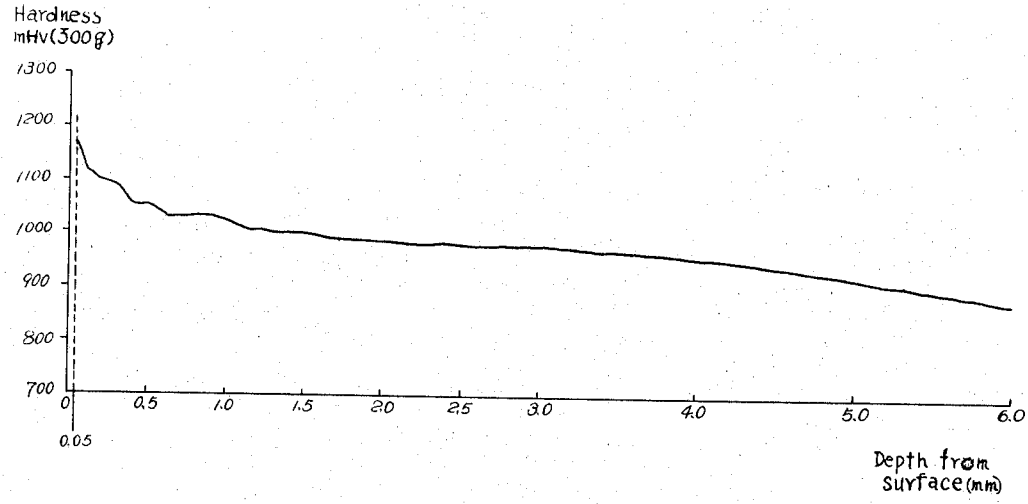
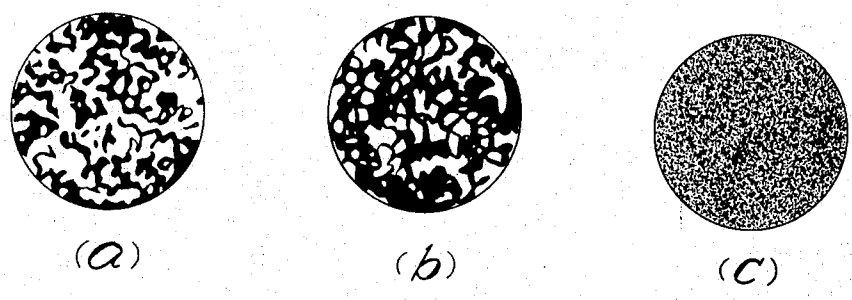


Fig. 3



INVENTOR
Toshimitsu Tanabe
BY
ATTORNEY

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3,362,854

SURFACE HARDENING TREATMENT OF STEEL OR ALLOY STEEL BY MEANS OF SUPERSONIC WAVE

Toshimitsu Tanabe, Tokyo, Japan, assignor to Kishichi Tanabe and Nobutsugu Utagawa, both of Tokyo, Japan, and Daniel J. Harnett, New York, N.Y.

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10 Claims. (Cl. 148—12.9)

The present invention relates to the surface hardening treatment of steel or alloy steel by means of supersonic waves.

More particularly, the present invention relates to the surface hardening treatment of steel or alloy steel through supersonic waves that impart thereto the properties of anti-abrasion, anti-fatigue, thermal resistance, pressure resistance and anti-corrosion. By immersing steel or alloy steel in a salt bath containing metallic powder of high mesh, or in a salt bath liberating carbon and nitrogen, with the vibration of the metallic structure produced by the supersonic wave as the main treatment and thermal energy as the medium thereof, the penetration and diffusion of various metallic properties are effected.

In the cementation of steel or alloy steel surface under conventional methods, the amount of thermal energy introduced thereto and the length of time for the treatment are the determining factors for the depth of diffusion or penetration which is roughly proportional to the energy plus time. Such treatment is subject to certain limitation in the degree and amount of heat the material under treatment can withstand without damaging its metallic and mechanical properties.

According to the present invention, the interstitial movement and substitution of atoms in metallic structure which are prerequisite for the penetration or diffusion of metallic properties can be activated by the supersonic wave, whereby the length of time for the treatment and the relative amount of thermal energy can be drastically reduced.

Through the proper adjustment of the supersonic wave frequency, the oscillation amplitude, and the activated cavitation created thereby, the transformation of metallic structure of an article under treatment is greatly accelerated because the cohesive concentration of metallic and other properties in the salt bath leads to the diffusion or cementation of the surface of the metal and towards the interior. The supersonic oscillations imparted to the salt bath generate therein cavitations which elevate potential energies of the ferro metals in said salt bath and simultaneously induct vibrations within the article which increase the speed of diffusion of said ferro metals into the article. The ferro metals contained in the bath scatter the supersonic oscillations within the bath and suppress the amplitude of standing waves created within the article by the vibrations, resulting in uniform diffusion of the ferro metals into the article. The ferro metals contained in the salt bath increase the acoustic impedance of the salt bath.

Reverting to the foregoing limitations of ordinary heat treatment, the depth of diffusion into the surface of metal

is proportional to the amount of thermal energy and the length of time for the treatment given thereto, consequently the depth of penetration is limited by considerations of grain growth, roughness of the surface, quenching cracks and fissures, and excessive distortion which usually resulted from high temperature, and the required long hours of treatment.

Taking rolls as an example, it is almost impossible under existing treatments to obtain more than 2 mm. of the diffused depth or the cemented layer on the surface of the rolls without coarsening the structure of the metal. However, in utilizing supersonic wave treatment according to the present invention, it is possible to obtain a depth of diffusion of 4 to 5 mm. and the hardness of more than mHv 1200 on the surface without causing the coarsening of the metallic structure. Moreover, the foregoing depth and hardness are obtainable with the temperature 15% lower and the length of time 80% less than the existing treatments require to obtain the same result. Furthermore, the application of supersonic wave according to the present invention has a great normalizing effect on metallic structure of the articles for treatment.

Tools and parts thus treated are greatly improved in quality, and their overall economic value significantly enhanced.

The following elements may be mixed in the salt bath under the present invention:

ferro-tungsten,
ferro-molybdenum,
ferro-titanium,
ferro-manganese,
ferro-silicon,
aluminium and chromium respectively in powdered form,
tungsten carbonate,
molybdenum carbonate,
titanium carbonate,
nickel carbonate,
chromium carbonate,
cobalt carbonate,
copper carbonate,
aluminium carbonate,
sodium cyanide,
sodium carbonate,
barium carbonate,
chromium chloride,
aluminium oxide,
barium chloride,
sodium chloride, and
ammonium chloride.

The transformation point of an article for the treatment varies according to the types of metal used, designs and the ultimate use as tools and parts, and a proportion of the above metallic and chemical elements that will constitute the components in the salt bath must be determined accordingly.

An article to be treated is immersed into the salt bath which contains a predetermined portion of various elements as above referred to; heated to a temperature in the range of 500° C.–1000° C., and supersonic waves are imparted thereto. Thereby the interstitial movement and substitution of atoms in the article thus being treated

are effected, accompanied by the normalizing effect of the metallic structure, and the metallic and chemical properties of the component are diffused into the metal, thus resulting in the formation of a cemented super hardness alloy surface layer. The tools and parts thus treated have increased fatigue life, anti-abrasion, anti-shock, anti-thermal and anti-corrosion qualities.

Referring to the accompanying drawings which illustrate one of the applications according to the present invention, FIGURES 1*a*, 1*b* and 1*c* show the supersonic wave oscillator, and the means by which it transmits the oscillation wave from the oscillation plate to the article under treatment, and the treatment in general. FIGURE 2 is a chart showing the hardness distribution of the article treated by the present invention. In FIGURES 3, (*a*), (*b*), and (*c*), the micro-photographs show the surface, intermediate zone and core of the article treated by the present invention.

In order to make the present invention practical, and produce good results, a proper selection and adjustment of frequency of supersonic wave and oscillation amplitude, composition of metallic and chemical elements, their grain size and purity, temperature and the length of time for the treatment, are the most important and determining factors.

The present invention permits the supersonic wave to be conducted directly to the article for treatment by connecting the oscillation plate to the article or indirectly by inserting the oscillation plate in the salt bath which contains the aforementioned compounds and is heated to a temperature in the range of 500°–1000° C.

The accompanying drawings FIGURES 1*a*, 1*b* and 1*c* show the article under treatment **6**, salt bath containing the compound **7**, salt bath case **8**, heater **9**, furnace body **10** and outer casing **11**.

The process and the order of the treatment according to the present invention are as follows:

The article to be treated **6** is either directly connected to the supersonic oscillation plate **5** as shown in FIGURE 1*a*, or receives the supersonic oscillation wave indirectly as shown in FIGURE 1*b*. The plate **6** is oscillated by the oscillation magnet coil **2** and the iron core **4** which works through the high frequency input **1'** connected to the high frequency oscillator **1**. The thermocouple **13** should be inserted into the salt bath **7** as shown in FIGURE 1*c* in order to facilitate automatic regulation.

After the foregoing treatment has been given, the article should be quenched and tempered according to the types of base metal used and their design, whereby a normalized surface layer containing rich metallic carbide is obtainable.

The depth of the cemented layer varies according to the base metal and depends upon the ultimate use as tools or parts, and is generally 4–5 mm. in depth including the effective zone.

In addition to the outstanding effects of the supersonic wave in the heat treatment of the present invention, the following characteristics should be noted:

(a) The supersonic wave completely agitates metallic and chemical elements in the bath, of different specific gravity and maintains a uniform distribution of elements in the salt bath without any mechanical mixing. At the same time, the agitation aforesaid maintains a uniform temperature throughout the salt bath;

(b) Deterioration of the salt often occurs due to residual moisture present in the salt itself and diffusion compound. The supersonic waves and the resulting effect of cavitation drives off moisture from the salt as well as the diffusion compound, thus avoiding the oxidation and decarborization of the article under treatment.

Examples of the treatment according to the present invention are as follows:

Example 1 (refer to FIGS. 1a and c)

Item: Sendjimir work roll.

Dimensions: 70 mm. diameter; 1200 mm. long.
Weight: 37 kg. (approximately).
Metal: High speed steel (M–50).
Composition: C=0.85, Si=0.35, Mn=0.4, Cr=4.5, Mo=4.5, V=1.0.
Supersonic wave frequency: 15 kc.
Frequency output: 5 kw.
Diffusion elements in the salt bath are as follows:

	Percent by weight
Ferro-tungsten (in powdered form)	5
Ferro-molybdenum	3
Tungsten carbonate	15
Molybdenum carbonate	15
Chromium carbonate	7
Cobalt carbonate	15
Powdered aluminium	3
Sodium cyanide	15
Sodium carbonate	15
Barium carbonate	7

The roll should be machined in the same manner as for any heat treatment and roughly polished (to a degree of 1S), and immersed into the salt bath containing the compound above referred to and heated to 950° C. for approximately 3 hours, simultaneously, supersonic waves are imparted to the roll through the oscillation plate secured to the upper portion of the roll.

Then, the roll should should be furnace-cooled. This is to obtain satisfactory diffusion of the elements and at the same time to remove the heat stress from the internal metallic structure.

After this procedure, conventional quenching and tempering which are suitable for high speed steel should follow. By the present invention, however, the secondary hardening will be supplemental to reduce the hardness difference, and inequality in the coefficient rate of expansion between the hard surface and core. The hardness curve shown in FIG. 2 and the microphotographs in FIG. 3 are for a roll treated by the present invention. In FIGS. 2 and 3, density of the carbide in the surface which gradually reduces towards the core is clearly shown and the hardness diminishes accordingly.

The hardness at the surface of the roll thus treated and combined with through hardening is mHv 1168 as shown in FIGS. 2 and 3, and it compares to the normal hardness of approximately mHv 850 which conventional hardening treatment will give to M–50 steel.

The hardness shown in FIG. 2 is given in figures in the following table.

Depth from surface(mm.)	Hardness, mHv (300 g.)	Depth from surface(mm.)	Hardness, mHv (300 g.)
0.05	1,168	2.50	982
0.10	1,139	3.00	976
0.20	1,110	3.50	966
0.30	1,110	4.00	966
0.40	1,051	4.50	919
0.50	1,051	5.00	919
0.60	1,036	5.50	890
0.70	1,036	6.00	876
0.80	1,036	7.00	876
0.90	1,032	8.00	976
1.00	1,020	9.00	823
1.50	990	10.00	823
2.00	982	----------	----------

Surface Hardness: HRA 87.5, mHv 1168.
Intermediate Zone Hardness: HRA 86, mHv 982.
Core Hardness: HRA 84.5, mHv 823.

The service life of the roll thus treated by the present invention is 5 to 10 times that of the roll conventionally treated.

The Sendjimir work roll is generally subject to the severest conditions among all the rolls, as in the case of pinch rolls, pressure rolls, leveller rolls, forming rolls, guide rollers etc., the present invention assures increase of service life of 10 to 100 times that of articles treated by conventional methods.

Example 2 (refer to FIGS. 1b–6)

Item: Trimmer knives for steel plate.

Dimensions: OD 406 mm. x ID 215 mm. x 38 mm. 3 pieces.

Weight: 28 kg. per piece×3=84 kg.

Metal: JIS Standard SKD–11 (die steel).

Composition: C=1.6, Si=0.4, Mn=0.5, Ni=0.5, Cr=13.0, Mo=1.2, V=0.5.

Supersonic wave frequency: 20 kc.

Frequency output: 5 kw.

Composition of the diffusion elements in the salt bath:

	Percent (in weight)
Ferro-molybdenum (in powdered form)	5
Ferro-tungsten (in powdered form)	3
Molybdenum carbonate	15
Tungsten carbonate	10
Chromium carbonate	8
Cobalt carbonate	8
Ammonium molybdate	11
Sodium cyanide	15
Sodium carbonate	15
Barium carbonate	7
Aluminium (in powdered form)	3

Method of the treatment is substantially the same as in Example 1, but the length of time for the treatment is 2 hrs. instead of 3 hours of Example 1 and a higher frequency is used. These differences are due to the fact that in Example 1 the oscillation plate was directly connected to the article as shown in FIG. 1*a*, while in Example 2 the oscillation plate is inserted into the salt bath as shown in FIG. 1*b*, whereby the salt bath itself is vibrated and the article is indirectly vibrated. Consequently higher frequency becomes necessary.

After the above treatment, the article is given the ordinary treatment of quenching and tempering, and the hardness obtainable on the surface is over mHv 1100, while the hardness of this type of metal under the conventional heat treatment is mHv 750.

The service life of the trimmer knives thus treated is 10 to 20 times longer than that of conventionally treated knives.

Example 3

Item: Roller and ball bearings.

Dimensions: Outer race—OD 100 mm., ID 82 mm.; thickness, 25 mm.; inner race—OD 65 mm.; ID 45 mm.: thickness, 25 mm.; ball dia., 25 mm.

Weight: 800 kg. per set, 20 sets at a time by the continuous treatment system.

Metal: JIS Standard SUJ—2 (bearing alloy steel) equivalent to SAE 52100.

Composition: C=1.1, Si=0.35, Mn=0.5, Cr=1.6.

Supersonic wave frequency: 30 kc.

Frenquency output: 2 kw.

The diffusion elements in the salt bath are substantially the same as in Example 2, but the temperature of the salt bath is maintained at 950° C. and the length of time for the treatment is 1.5 hrs. The higher frequency is required in this case than that of previous treatment, because the bearing parts of different dimensions are treated together in the same salt bath and a more powerful oscillation is necessary to obtain uniform surface layer and the hardness in all pieces.

After the above treatment, the bearing parts are quenched and tempered in the usual or normal way. The hardness of the parts after the treatment of the present invention combined with through hardening is mHv 1100 which compares to a normal hardness of mHv 750 for the same steel when hardened conventionally.

Dimensional accuracy of normally 4 or 5 microns is required as the maximum tolerance for bearing parts, and they are no longer serviceable as bearings when 20 microns (max.) have been lost. Except in bearings for special purposes, the wear is generally caused by pitting as a result of rolling contact. In order to give higher fatigue life to the bearing parts, a high hardness and sufficient depth of the hardness are most desirable. Variation of lubricant viscosity and ambient temperature of the bearing in service should be considered separately.

A fatigue life test of a roller bearing after treatment by the present invention and performed by a certain bearing company in the United States showed a remarkable increase of the life as shown below:

Test bearing: outer diameter 2.560", bore 1.930", cylindrical bearing.

Steel: SAE 52100 bearing alloy steel.

Load: 7000 pounds.

R.p.m.: 1750 shaft rotation.

(With normal 52100 alloy steel, the bearing is expected to have a basic dynamic load rating of 12,300 pounds which will yield a rating life of 63 hours. The average life is estimated at 313 hours. Normal test will vary slightly from batch to batch but always within the range of 268 to 399 hours.)

The test under the conditions above referred to for the bearings treated by the present invention was performed for a series of 10 and the life obtained therefrom was within the range of 1800 to 2000 hours.

Example 4

The present invention can be effectively incorporated into some of the conventional surface treatments and can reduce the length of time for treatment considerably, yet obtain the same or better results, as shown below:

(A) *Liquid carburizing.*—A 1" square test piece made of JIS Standard SNCM–22 carburizing steel was heated at 935° C. for 2 hours in the salt bath containing equal quantity of sodium chloride and barium chloride and to which a supersonic wave of 15 kc. frequency and 2 kw. output was imparted. After the through hardening process of quenching and tempering, the said piece has 2 mm. of cemented layer and the hardness of mHv 850. This compares to 6 hours of heat treatment and the hardness of approximately mHv 700 which conventional carburizing requires.

(B) *Nitriding.*—A tapered drill of 1" diameter made of high speed steel, JIS Standard SKH–9, was heated at 450°–600° C. in the salt bath containing 30% sodium cyanide, 17% caustic soda, 30% sodium chloride and 23% caustic potash. After the hardening process of quenching and tempering, the said drill was subject to supersonic wave, frequency 15–20 kc. and output 2 kw., for 4 hours. The drill thus obtained had a 0.1 mm. cemented layer and hardness of mHv 1000. If the supersonic wave referred to above had not been imparted, it would require heat as much as 5 times longer than it did to obtain the same depth of cemented layer and hardness.

(C) *Chromizing.*—A test piece, 25 mm. x 50 mm. square, made of JIS Standard SK–5 alloy steel, was heated at 950° C. in the salt bath containing 45% chrome chloride, 30% barium chloride, 20% sodium chloride and 5% powdered chromium, to which a supersonic wave of 15 kc. frequency and 2 kw. output was imparted for 5 hours. The test piece was then furnace cooled at 900° C. for 4 hours, and obtained the cemented layer of 0.2 mm. and the hardness of mHv 1000.

(D) *Calorizing.*—A test piece, 5 mm. x 100 mm. square, made of 0.5 carbon alloy steel, was heated at 800° C. for 3 hours in the salt bath containing 50% powdered aluminum, 45% aluminum oxide and 5% ammonium chloride, to which a supersonic wave of 15 kc. frequency and 2 kw. output was imparted. The piece obtained had a cemented layer of 0.2 mm. and hardness of mHv 550.

In addition to the foregoing applications, the present invention can prove to be exceedingly effective for increasing tool life or fatigue life of knives, punches and dies of various kinds, and machine parts made of alloy

steel including austenic stainless steel which shall be treated in similar way to those stated in Examples 1, 2 and 3, resulting in greatly improved properties of anti-wear, anti-fatigue, anti-thermal, anti-shock and anti-corrosion properties.

Those examples stated herein are by way of example only and should not be considered as limiting the present invention. It can be effectively, practically and economically applied to any tools, tool parts, machine parts and similar objects made of steel or alloy steel, for which improvement of metallic and mechanical properties are desired.

What I claim is:

1. The method of surface hardening an article of steel or alloy steel, comprising, immersing said article in a molten salt bath containing metallic powder capable of liberating carbon and nitrogen and heated to from about 450° C. up to below the melting point of said article, while imparting supersonic vibrations to said article.

2. The process of surface hardening an article of steel or alloy steel, comprising, immersing the article in a molten salt bath capable of liberating carbon and nitrogen when heated, imparting supersonic vibrations to said article for a period of about 1.5 to 5 hours, while maintaining the temperature of the bath at about 450° C. to 950° C., and subsequently quenching and tempering the article.

3. The process of surface hardening an article of steel or alloy steel, comprising, immersing the article in a molten bath consisting of major amounts of tungsten carbonate, molybdenum carbonate, chromium carbonate, cobalt carbonate, sodium cyanide, sodium carbonate and barium carbonate, each about 7% to 15% by weight, and minor amounts of ferro-tungsten, ferro-molybdenum, and powdered aluminum, each about 3% to 5% by weight, all said materials being in powdered form, and imparting to the article while so immersed, supersonic vibrations of about 15 to 30 kc./sec., for a period of 1.5 to 5 hours, while maintaining the bath at a temperature of about 800° C. to 950° C.

4. The process of surface hardening an article of steel or steel alloy, comprising, immersing the article in a molten salt bath containing powder having major amounts of an ingredient selected from the group consisting of tungsten carbonate, molybdenum carbonate, cobalt carbonate, sodium cyanide, and sodium carbonate, and minor amounts of an ingredient selected from the group consisting of ferro-tungsten, ferro-molybdenum, chromium carbonate and barium carbonate, and imparting to the article supersonic vibrations at a frequency of about 15 to 30 kc./sec., for a period of about 1.5 to 3 hours, while maintaining the bath and article at a temperature of about 950° C.

5. A process of surface hardening an article of steel or alloy steel by cementation and diffusion comprising immersing said article into a molten salt bath which is heated at a temperature of above 450° C. but below the melting point of said article and which contains a major portion of sodium chloride, sodium cyanide and barium chloride to maintain proper fluidity and to avoid excessive deterioration of said salt bath, a minor portion of powders selected from the group consisting of ferro-tungsten, ferro-molybdenum, ferro-titanium and ferro-chrome, and a minor portion of chromium carbonate and cobalt carbonate; and imparting into said salt bath supersonic oscillations of more than 15 kc., said supersonic oscillations imparted to the molten salt bath generating therein cavitations which elevate potential energies of the ferro metals in said salt bath and simultaneously induct vibrations within the article which increase the speed of diffusion of the ferro metals into the article, said ferro metals contained in the molten salt bath scattering said supersonic oscillations within said bath and suppressing the amplitude of standing waves created within the article by said vibrations, resulting in uniform ferro metal diffusion into said article.

6. The process of claim 5 for surface hardening an article of steel or alloy steel through cementation and diffusion wherein said salt bath contains more than 60 weight percent of sodium chloride and sodium cyanide and has a temperature of from 450° C. to 750° C.

7. The process of claim 5 for surface hardening an article of steel or alloy steel through cementation and diffusion wherein said salt bath contains more than 70 weight percent of sodium chloride, sodium cyanide and barium chloride and has a temperature of from 750° to 950° C.

8. A process of surface hardening an article of steel or alloy steel by cementation and diffusion comprising immersing said article into a molten salt bath which is heated at a temperature of above 450° C. but below the melting point of said article and which contains a major portion of sodium chloride, sodium cyanide and barium chloride to maintain proper fluidity and to avoid excessive deterioration of said salt bath, a minor portion of powders selected from the group consisting of ferro-tungsten, ferro-molybdenum, ferro-titanium and ferro-chrome, and a minor portion of chromium carbonate and cobalt carbonate; and imparting to said article supersonic oscillations of more than 15 kc., said supersonic oscillations imparted to the article generating vibrations within the article which increase the speed of diffusion of the ferro metals into said article, and said ferro metals contained in said salt bath increasing the acoustic impedance of said salt bath and suppressing the amplitude of standing waves generated within the article by said vibrations, resulting in uniform diffusion of said ferro metals into said article.

9. The process of claim 8 for surface hardening an article of steel or alloy steel through cementation and diffusion wherein said salt bath contains more than 60 weight percent of sodium chloride and sodium cyanide and has a temperature of from 450° to 750° C.

10. The process of claim 8 for surface hardening an article of steel or alloy steel through cementation and diffusion wherein said salt bath contains more than 70 weight percent of sodium chloride, sodium cyanide and barium chloride and has a temperature of from 750° to 950° C.

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DAVID L. RECK, *Primary Examiner.*

RICHARD O. DEAN, HYLAND BIZOT, *Examiners.*