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PROCESS OF COATING AND HOT WORKING OF METALS

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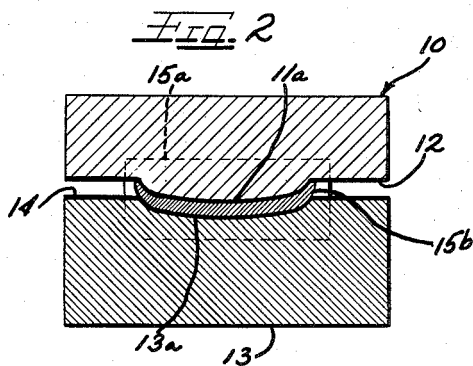
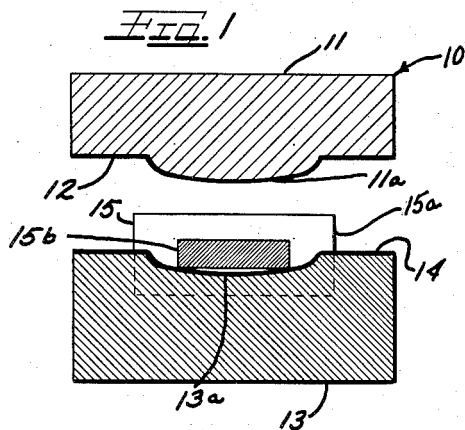


FIG. 3

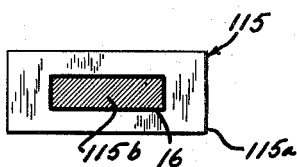


FIG. 4

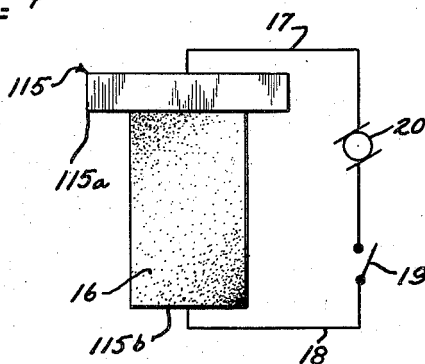


FIG. 5

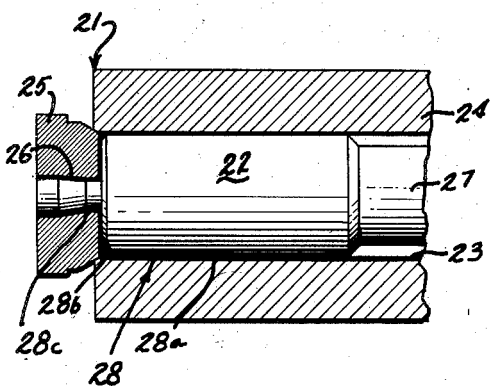
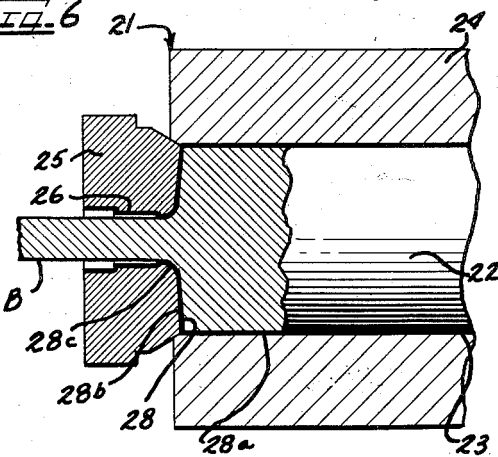


FIG. 6



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PROCESS OF COATING AND HOT WORKING OF METALS

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18 Claims. (Cl. 29—424)

The instant invention relates to metal working processes, metal working dies for use therein, and methods of making such dies, and more particularly, the instant invention relates to metal working processes wherein a refractory coating is applied between the surface portions of the die and a preheated workpiece, and to metal working dies having such coating thereon and a method of preparing the same.

In metal working processes, the metal workpiece is subjected to heat and pressure, by the application of dies, in order to form the workpiece into the ultimate shape desired. The appropriate surface portions of the dies are applied to the workpiece under very great pressures such as to generate heat and the workpiece is ordinarily preheated to an appreciable extent to make it more easily deformable under the pressures employed. Lubricants have been suggested heretofore for use in the various metal forming or metal working processes which include stamping, cutting, swaging, rolling, forging and piercing. Such lubricants are intended to diminish the friction between the workpiece and the dies so as to avoid local defects in the formed piece and to minimize wear and tear on the dies themselves. Also the lubricants are intended to diminish the total pressure necessary in effecting the work and perhaps facilitate separation of the dies and the work at the end of the forming operation. It is also desirable to have lubricants which afford a certain amount of insulation between the dies and the metal workpiece so as to lower as much as possible the temperature of the dies and preserve the original metallurgical properties of the dies. Excessive surface heating of the dies may also tend to cause seizure.

The lubricants heretofore employed include oils and fats of mineral, vegetable or animal origin which may also contain other bodies such as graphite, talcum or the like. These materials all leave something to be desired in that they do not afford sufficient protection to the dies so as to minimize wear and tear of the dies and to maintain lower temperatures in the dies. Also, the oils or fats are not highly refractory and decompose at metal working temperatures.

More recently, other lubricants of a ceramic character have been suggested. For example, Sherman United States Patent No. 2,430,083 suggests the application of enamel to the surfaces of the die and/or the workpiece, using ceramic or porcelain enamel compositions which are applied to the surfaces of the workpiece and/or the dies and fired thereon to provide a typical porcelain enamel coating. This coating is made of ceramic enameling material which melts and fuses during the forging operation and Sherman teaches the use of his invention particularly in connection with a single stroke forging operation.

Sejournet United States Patent No. 2,538,917 discloses a metal extrusion process wherein layers of glass (glass fibers, slag or other suitable fusible ceramic) are positioned against the back of the extruding die between

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the workpiece and the die so that the glass will fuse to afford a protective film. One of the principal difficulties with both the Sherman and the Sejournet operations is that the lubricant is consumed during the operation and must be replaced repeatedly, either by placing additional glass disks in the Sejournet device or by applying enamel and firing to fuse the same in the Sherman device.

The above identified Sejournet patent also mentions (column 5, lines 6 et seq.) that a lubricant can be employed between the workpiece and the container which surrounds the workpiece as it is urged toward the die; and this lubricant may include materials which remain solid at extrusion temperatures. Such lubricants include talc and aluminum silicate which may be employed in the form of very fine powders and this also leaves something to be desired because the powders must be replaced from time to time and, of course, cannot be used very effectively at all in a forging operation for example.

The instant invention provides a unique solution to the problem of providing a suitable lubricant-insulator for metal working processes. According to the instant invention, a coating of refractory material is applied to the workpiece and/or dies in the form of an adherent refractory material, comprising predominantly if not entirely a refractory metal oxide, which remains refractory or solid at the metal working temperatures and possesses sufficient impact resistance, flexibility and adherence to the metal surface so as to afford protection for the dies in the manner hereinbefore described as desirable during repeated or continuous use of the dies. The instant refractory coatings are not fused and, therefore, consumed during the metal working process, but remain solid and adherent to the die surfaces during repeated or continuous use of the dies.

It is, therefore, an important object of the instant invention to provide an improved metal working process, an improved die for use therein and an improved method of coating or protecting said die with a lubricant-insulator of permanent or lasting characteristics.

It is a further object of the instant invention to provide an improved metal working die having an adherent coating thereon which is refractory at metal working temperatures and which insulates the die from the workpiece; and an improved method of applying a refractory coating to such die.

Still a further object of the instant invention is to provide an improved metal working process which comprises applying such dies to a workpiece under pressure to form the piece.

Other objects, features and advantages of the present invention will become apparent to those skilled in the art from the following detailed disclosure thereof and the drawings attached hereto and made a part hereof.

On the drawings:

Figure 1 is a sectional elevational view showing forging dies separated with a workpiece in location therebetween;

Figure 2 is a view similar to Figure 1 showing the dies almost in closed position substantially at the completion of the forging step;

Figure 3 is a sectional elevational view of a modified form of workpiece adapted for use with the dies of Figures 1 and 2, having a refractory coating thereon;

Figure 4 is a top plan view of the modified workpiece of Figure 3 with parts shown diagrammatically in connection with an electric circuit attached to the workpiece of Figure 4;

Figure 5 is a sectional elevational view showing in part the container and die of an extrusion press with the workpiece in position; and

Figure 6 is a view similar to Figure 5 showing the workpiece partially extruded through the die.

As shown on the drawings:

In Figures 1 and 2 the die assembly indicated generally by the reference numeral 10 comprises an upper movable male die 11 presenting a working face 11a that is coated with a refractory layer 12 in accordance with the instant invention (in a manner which will be described hereinafter); and a lower fixed female die 13 presenting a working face or surface portion 13a corresponding to the working face or surface portion 11a of the mating upper die 11. The working faces 11a and 13a are both coated with refractory layers 12 and 14, respectively, in accordance with the instant invention.

In the practice of the instant invention a workpiece 15 having a base or root portion 15a and a blank portion 15b to be deformed is heated to forging temperature (for example, a steel piece 15 heated to about 2000-2400° F. or 1100-1300° C.) is positioned in the cavity of the female die 13, with the blank portion 15b resting in the cavity. The movable top die 11 is then brought down against the deformable portion 15b' (as shown in Figure 2) to deform the same into the desired shape. In the normal operation, it may require a series of hammering blows by the movable upper die 11 in order to finally forge the blank portion 15b' into the shape desired. The coatings 12 and 14 provide a lubricant-insulator coating for the dies 11 and 13, respectively, so that the working faces 11a and 13a, respectively, are not heated as nearly to the forging temperature as would result if the coatings 12 and 14 were eliminated. The coatings 12 and 14 do not fuse during the forging operation and are not shattered by the impact of hammering blows by the movable die 11. When the piece 15 has been forged, it may be easily removed from the female die 13 and another workpiece may be placed therein.

In Figures 3 and 4 there is shown a workpiece 115a also having a root portion 115a and a blank deformable portion 115b. The blank deformable portion 115b is provided with a coating 16 which is the same type of coating as the coatings 12 and 14 previously described. As shown in Figure 4, opposite ends of the workpiece 115a may be connected to leads 17 and 18 which in turn are connected through a switch 19 to a suitable source 20 of a heating electric current which can flow through the piece 115 and heat the same. Because of the highly refractory properties of the coating 16, the piece 115 may be heated up to substantially higher than the ordinary forging temperatures, or at least to forging temperatures in the higher range, so that the workpiece 115 can be very readily forged, for example, by heating to 1300° C. Because of the electrical insulating properties of the coating 16, the piece 115 could actually be placed in the female mold 13 and heated in this manner. The refractory-insulating properties of the coating 16 and/or the coating 14 serve to protect the mold 13 from thermal degradation of the metallurgical properties thereof, thereby permitting the use of maximum forging temperatures.

Referring now to Figures 5 and 6, it will be seen that the metal extrusion assembly, indicated generally by the reference numeral 21 includes a steel workpiece 22 suitably positioned in an extrusion chamber 23 formed by a container 24 closely surrounding the workpiece 22. A die 25 is located at one end of the extrusion chamber 21 and has a die opening 26 through which the bars are to be extruded by pressure applied to the workpiece 22 by a punch 27. Actually, the container 24 and the die proper 25 are both die members in that they serve to shape the workpiece 22, the container 24 holds the workpiece 22 against lateral movement, while the die 25 effectively reduces the cross-sectional area of the workpiece 22. A coating 28 is applied along the working face of the container 24, at 28a, along the back of the die 25, at 28b, and at the mouth of the die 25 or at the die opening 26, as at 28c. The coating 28 is the same type of coating as the coatings 12, 14 and 16 herein-

before described; and this coating remains adherent to the container walls during the operation and effectively insulates the container against the extremely high heat generated by the extrusion of the workpiece through the die opening 26 to form the bar B shown in Figure 6. The coating 28 adheres to the surface of the walls of the container 24 and adequately lubricates these walls as well as insulating the walls. The coating 28 also provides a lubricant-insulator for the back of the die 25, while coating 28b remains solid during the extruding process and does not flow out the die opening 26 with the metal. The coating 28c in the die opening 26 also remains solid during the extrusion process and provides a lubricant-insulator for protecting the die 25 at this critical region.

A key to the instant invention resides in a process which comprises directing a high temperature flame at the working face of a metal working die and injecting into said flame certain particulate materials which deposit a refractory coating on said face, such as the coatings 12, 14, 16 and 28 hereinbefore described. It has been found that remarkably improved coatings result when the material injected into the flame is a mixture of a refractory metal oxide and a fluoride of an alkali metal (e. g. lithium, sodium, potassium, etc.) or an alkaline earth metal (e. g. magnesium, calcium, strontium, or barium).

The coatings of the present invention are remarkably superior to those produced by applying various glass compositions, even with the flame spraying method here employed, onto the base. Apparently, the difference in atomic structure between the coatings produced according to this invention and those produced by spraying fused glass onto the same objects accounts for this wide variation in properties. As is well known, glass compositions are amorphous in structure while it has been determined that the atomic structure of the coatings of the instant invention, as evidenced by X-ray diffraction data, are crystalline. It is believed that the flame spraying operation when carried out according to the present invention results in an in situ formation of a reaction product between the refractory metal oxide and the fluoride. Apparently, the fluoride replaces at least some of the oxygen atoms in the crystal lattice of the oxide to produce an oxy-fluoride.

In general, as the oxide content of the coating increases, the thermal shock resistance and the insulating properties are also increased; whereas the addition of the fluoride, with subsequent formation of the oxy-fluoride, appears to increase substantially the bonding ability of the oxide coating without significantly affecting its refractory properties or its insulating properties.

Relative proportions between the oxide and the fluoride will vary considerably, depending upon the particular system employed. In some systems, the fluoride may be present in very minor amounts such as about 1% of the refractory composition (unless otherwise stated herein, all percents being percent by weight). In other cases, the fluoride content may be as high as 25%, or even higher. Generally, however, the fluoride will constitute about 2 to about 20% of the composition with the balance being the oxide. Since refractory properties are of particular importance in the instant invention, the fluoride content is preferably within the range of about 2 to about 10%; and in the case of certain refractory metal oxides, particularly alumina, it has been found that an effective refractory coating can be obtained using minimum amounts of the fluoride (i. e. less than 2%) or even by omitting the fluoride, as will be explained hereinafter in this disclosure. In all cases, however, the incorporation of the fluoride in the composition imparts distinctly superior properties.

The instant coatings are prepared by injecting particulate material of the type herein described in a flame having a flame temperature within the range of about

1700° C. to about 3500° C. Preferably this is accomplished by injecting the particulated material into an oxygen stream, mixing the stream with acetylene or hydrogen, and then burning the stream. The nozzle of the flame spraying apparatus is held at a distance varying from 1 to 6 inches from the object to be coated or the die surface. The extremely hot zone of the flame is, of course, spaced some distance from the surface and by careful adjustment of the flame to achieve a temperature most suitable for particular particulated mixtures it is possible to apply an adherent coating to the die surface without effecting any appreciable heating of the die surface so as to alter the metallurgical properties thereof. A feature of the instant invention resides in the fact that it provides for the application of refractory coatings to the metal surface of the die, using refractory materials which have fusion temperatures substantially above the actual melting temperature of the metal itself. In actual operation the gas components are fed into the nozzle to create a flame having a temperature within the range just mentioned and the particulated material is fed into the flame at a rate sufficient to carry out reasonably rapid coating of the object but not so great as to disturb the operation of the flame and/or result in the formation of a loose or non-adherent coating. The adequacy of the coating can be readily observed by the operator after a small amount of practice and the optimum coating conditions can be obtained by making minor adjustments in the gas mix going to the flame and the rate of feed of the particulated material, while observing the actual properties of the coating being applied. Varying the distance between the nozzle of the flame and the object may also be used to arrive at optimum coating conditions.

One system which provides an extremely satisfactory coating is the combination of alumina and magnesium fluoride, and most preferably alpha-alumina monohydrate and magnesium fluoride. This system produces coatings which are harder and more adherent than those produced with other fluoride binders. This is believed to be due to the fact that alumina and magnesium fluoride have the same type of crystal structure and are more compatible than combinations of alumina and other fluorides. Alumina admixed with from about 2 to about 5% magnesium fluoride has been found to be particularly suitable. For example, coatings were made using alumina containing 3% magnesium fluoride (and the balance alpha-aluminum monohydrate) having an average particle size of about 200 mesh; and coatings having thicknesses up to 50 mils and beyond could be applied with ease. An X-ray analysis of the coating showed it to have the gamma type alumina structure. The hardness of the coating was found to be about the same as that of topaz or emerald and the coatings had excellent scratch resistance, in that they could not be scratched with a hardened steel needle. These coatings also possess extremely good refractory and insulative properties and impact resistance. The coatings have been successfully used on forging dies and other metal working dies as lubricant-insulators in the manner shown in the previous disclosure relating to the drawings (wherein the coatings 14, 16 and 28 are shown in disproportionately great thickness).

Another system which provides particularly good coatings involves the combination of oxides of metals of group IVA (titania, zirconia and hafnia) and alkaline earth metal fluorides (particularly magnesium and calcium fluorides). In particular, zirconia compositions containing about 2 to about 5% calcium fluoride have been found to be particularly suitable for continuous high temperature use (such as in the case of the coating 28 in Figures 5 and 6). Coatings made from 3% calcium fluoride and the remainder zirconia, using an average particle size of about 200 mesh, and using the operating conditions hereinbefore described, were applied in thicknesses of

about 10 mils and these coatings were found to have extremely good resistance to high temperatures. The coatings, upon X-ray analysis, have been found to be a stabilized isometric form rather than the relatively unstable monoclinic form.

The instant coatings may also be improved in certain cases by the incorporation of from about 5 to about 15% of aluminum powder in the composition injected into the flame (which presumably is converted to alumina during the flame spraying). For example, a composition containing 10% calcium fluoride, 10% aluminum powder, and the balance zirconia was found to give a coating which exhibited remarkable resistance to heat.

In general, coatings may be applied ranging from 1 micron to 0.050 inch in order to obtain certain advantages of the invention, but it is preferable to employ coatings within the range of about 5 to about 50 mils for the purposes here involved.

In addition to the foregoing, the following systems have been found to produce satisfactory flame sprayed ceramic coatings:

Table I

	TiO ₂ —MgF ₂
	MgO—LiF
25	Al ₂ O ₃ , MgO—MgF ₂ , LiF
	Al ₂ O ₃ —MgF ₂ , CaF ₂ , Mg ₂ P ₂ O ₇ , and/or Mg(C ₂ H ₃ O ₂) ₂
	Al ₂ O ₃ , Na ₂ SiO ₃ —NaBF ₄
	MgO—MgF ₂ , LiF
	CaO—KF
30	ZrO ₂ —LiF, CaF ₂ , and/or Li ₂ Al ₂ O ₄
	ThO ₂ —CaF ₂
	UO ₂ —CaF ₂
	CeO ₂ —CaF ₂
	Pr ₂ O ₃ —CaF ₂

In connection with the foregoing, it is not essential that the starting material be an oxide itself, but only that the compound employed be decomposed under the conditions of flame spraying to produce the oxide. For example, magnesium carbonate can be employed as a suitable source of magnesium oxide.

The equipment used to apply the flame sprayed coating may take any of a variety of forms. Most simply, an oxyacetylene cutting torch can be modified by the inclusion of a venturi nozzle to permit injection of the finely divided particulated mixture of the oxide and the fluoride into the flame while the flame is directed at the object to be coated.

Better results are secured in the process of the present invention if the powdered mixture fed into the flame is homogeneous, and has a particle size less than about 100 mesh. Substantial increases in spraying efficiency of the coatings can be achieved by pre-sintering the mixture prior to its injection into the flame. In this technique, the mixed powders are pressed into pills or bars, and the compacts which result are heated to temperatures in the neighborhood of the melting point of the fluoride. At this temperature, some reaction may take place between the oxide and the fluoride to produce the oxyfluoride. The sintered product is then ground into powder having a particle size less than about 100 mesh for injection into the flame spraying apparatus. When the material is pre-sintered, it has been found to produce coatings which are superior to simple mixtures of the powders not only in their mechanical properties, but also in respect to porosity and corrosion resistance.

The porosity of the coatings produced can be reduced by including a powdered metal such as aluminum in the flame spraying composition. Generally, from about 2% to about 30% of the metal will be effective to seal up the pores of the coating at the operating conditions of the spraying apparatus.

Another important aspect of the instant invention resides in the fact that certain refractory metal oxides such as alumina may be flame sprayed using the instant process in the absence of a fluoride so as to obtain a highly sat-

isfactory lubricant-insulator coating on a metal working die (or on the metal workpiece, if such is desired). Apparently such metal oxides undergo certain chemical or "crystal" chemical changes during the flame spraying operation so as to develop highly suitable properties. The reaction undergone using only the oxide is doubtlessly different from the reaction apparently undergone using the oxide in combination with the fluoride (which has been indicated as producing better results). The instant invention affords the additional advantage of employing such oxides as alumina in the flame spraying process. For example, using the flame spraying conditions hereinbefore described and alpha-alumina monohydrate in particulate form, having an average particle size of about 200 mesh, it is found that a coating of about 25 mils applied to a die provided a very sturdy and adherent lubricant-insulator coating, which stood up extremely well during forging operations.

Still another aspect of the instant invention resides in the fact that certain refractory metal oxides, particularly zirconia, may be combined with various lithium compounds in the practice of the instant invention to obtain distinctly superior results. As indicated in Table I, oxides such as magnesia and alumina may be combined with lithium compounds such as lithium fluoride to obtain advantageous results in the practice of the instant invention. Zirconia may also be combined with lithium fluoride to obtain advantageous results; but the combination of zirconia and lithium aluminate ($\text{Li}_2\text{Al}_2\text{O}_4$). As in the case of each of the systems hereinbefore described, as well as the instant system, the zirconia employed is preferably of the "stabilized" type. Stabilized zirconia is an article of commerce, usually containing from about 3 to 6% by weight of calcium oxide or magnesium oxide.

The proportions employed in the coating composition may vary, depending upon the nature of the coating desired. In most cases, however, the lithium aluminate will be present in amounts from about 0.001 to about 0.1 times the amount of zirconia present in the mixture being flame sprayed. Zirconia compositions containing about 2 to about 5% lithium compounds, such as lithium fluoride or lithium aluminate, have been found to be particularly suitable for high temperature use. The instant coating composition may consist simply of the powdered mixture of lithium aluminate and zirconia, the particles having a size less than about 100 mesh; but improved results are obtained and a small amount of the zirconia is replaced by calcium fluoride. The zirconia preferably contains about 2 to about 5% calcium fluoride prior to mixing the zirconia with the lithium aluminate. In order to secure a more homogeneous mixture of the calcium fluoride and the zirconia, it is preferable to blend the two compounds and to fire the mixture at a temperature between 1500° F. and the melting point of the fluoride. Apparently some reaction takes place under these conditions, perhaps resulting in the production of the oxy-fluoride. The product is then ground into a powder having a particle size less than about 100 mesh for injection into the flame spraying apparatus with the similarly sized lithium aluminate. The resulting coating sprayed in accordance with the procedures hereinbefore set forth are the thickness of about 25 mils has superior adherence and resistance to high temperatures and the die so coated stands up extremely well during forging operations.

It will be understood that modifications and variations may be effected without departing from the novel concepts of the present invention.

I claim as my invention:

1. A process which comprises directing a high temperature flame at the working face of a metal working die and injecting into said flame particulated alumina to coat said face, heating a workpiece to metal working temperatures, and applying the working face of the die to said piece to form the same.

2. A process which comprises directing a high tem-

perature flame at the working face of a metal working die and injecting into said flame a particulated mixture of refractory metal oxide and a fluoride of a metal selected from the group consisting of alkali metals and alkaline earth metals to coat said face, heating a workpiece to metal working temperatures, and applying the working face of the die to said piece to form the same.

3. A process which comprises directing a high temperature flame at the working face of a metal working die and injecting into said flame particulated alpha-alumina monohydrate to coat said face, heating a workpiece to metal working temperatures, and applying the working face of the die to said piece to form the same.

4. A process which comprises directing a high temperature flame at the working face of a metal working die and injecting into said flame a particulated mixture of alpha-alumina monohydrate and magnesium fluoride to coat said face, heating a workpiece to metal working temperatures, and applying the working face of the die to said piece to form the same.

5. A metal working die comprising a body and a working face coated by directing thereat a high temperature flame and injecting into said flame particulated alumina.

6. A metal working die comprising a body and a working face coated by directing thereat a high temperature flame and injecting into said flame particulated alpha-alumina monohydrate.

7. A metal working die comprising a body and a working face coated by directing thereat a high temperature flame and injecting into said flame a particulated mixture of refractory metal oxide and a fluoride of a metal selected from the group consisting of alkali metals and alkaline earth metals.

8. A metal working die comprising a body and a working face coated by directing thereat a high temperature flame and injecting into said flame a particulated mixture of alpha-alumina monohydrate and magnesium fluoride.

9. A method of making a metal working die that comprises directing at the forging face thereof a high temperature flame and injecting into said flame particulated alumina.

10. A method of making a metal working die that comprises directing at the forging face thereof a high temperature flame and injecting into said flame particulated alpha-alumina monohydrate.

11. A method of making a metal working die that comprises directing at the forging face thereof a high temperature flame and injecting into said flame a particulated mixture of refractory metal oxide and a fluoride of a metal selected from the group consisting of alkali metals and alkaline earth metals.

12. A method of making a metal working die that comprises directing at the forging face thereof a high temperature flame and injecting into said flame a particulated mixture of alpha-alumina monohydrate and magnesium fluoride.

13. A metal working process that comprises heating a metal workpiece and applying surface portions of dies under pressure to surface portions of the piece to form the piece, in the presence of a coating applied to one of said surface portions by directing thereat a high temperature flame and injecting into said flame a particulated mixture of zirconia and calcium fluoride.

14. A metal working process that comprises heating a metal workpiece and applying surface portions of dies under pressure to surface portions of the piece to form the piece, in the presence of a coating applied to one of said surface portions by directing thereat a high temperature flame and injecting into said flame a particulated mixture of zirconia and lithium aluminate.

15. A metal working die comprising a body and a working face coated by directing thereat a high temperature flame and injecting into said flame a particulated mixture of zirconia and calcium fluoride.

16. A metal working die comprising a body and a

working face coated by directing thereat a high temperature flame and injecting into said flame a particulated mixture of zirconia and lithium aluminate.

17. A metal working process which comprises heating a metal workpiece and bringing said workpiece into contact with a coated surface of a forming member, said coated surface being integrally bonded to said forming member and consisting of a flame sprayed sintered refractory composition having a melting temperature in excess of the temperature at which said workpiece is to be shaped.

18. A metal working process that comprises heating a metal workpiece and applying surface portions of dies under pressure to surface portions of the piece to form the piece, in the presence of a coating applied to at least one of said surface portions of said dies by directing thereat a high temperature flame and injecting into said flame particulated alumina, said coating being refractory at the workpiece temperature.

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