

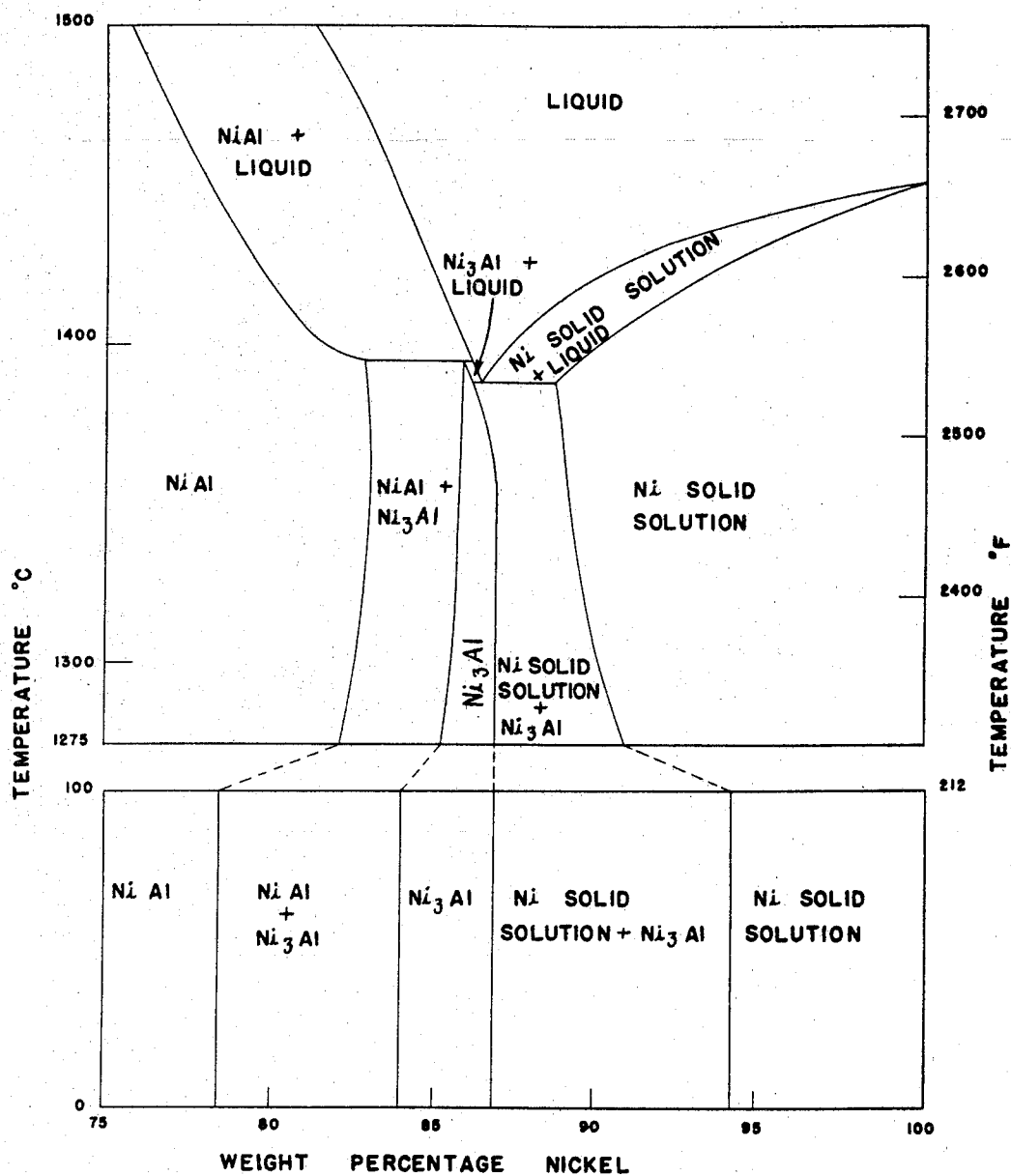
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METHOD OF MAKING Ni_3Al

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METHOD OF MAKING Ni_3Al

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The present invention deals with a preparation of improved metallic compositions of good oxidation resistance and having a high degree of workability.

The compositions of the present invention include what may be termed "intermetallic compounds" in the sense that the predominating ingredient in the compositions represents a combination of two metals in definite molecular proportions. The empirical formula for the preferred compound of the present invention is Ni_3Al . We have found that this compound has extraordinary oxidation resistance properties, and excellent hot strength properties, as well as being workable.

In order to understand the structure of the compositions of the present invention, some discussion should be made of theory of solid solutions and the various types of combinations which two or more metals may undergo when combined in the form of a solid solution. It has long been established that in many alloy systems, the addition of one metal to another does not produce a new phase, but results in the formation of a homogeneous alloy which is the result of a solid solution of one metal in the other. The solid solutions which form the end phases in equilibrium diagrams are called primary solid solutions having the same structure as the parent metal from which the solid solution is formed. Under conditions of solid solution, two distinct types of solid solutions are possible. The first of these, the more general case, is that of a substitutional solid solution in which the solute atoms replace those of the solid, so that the two different atoms are situated on a common lattice. The other type of solid solution is the interstitial solid solution in which the solute atoms fit into the spaces between those of the solvent. This latter case occurs when there is a great difference in size between the atoms making up the solid solution, and occurs most frequently when small atoms such as carbon, nitrogen, boron, and hydrogen are combined with metal atoms having a substantially larger size. Ferrite, austenite, and the hard metal carbides, are good examples of interstitial solid solutions.

As the size of the atoms making up the solid solution becomes more nearly equal, a substitutional solid solution results. In most substitutional solid solutions, the different atoms are arranged at random on a common lattice, but in some cases, alloys which at high temperatures consist of random substitutional solutions undergo an atomic rearrangement upon slow cooling or annealing, resulting in the different atoms taking up regular positions in the crystal lattice and thus forming an ordered structure. The basic crystalline structure is, however, the structure of the parent solid solution. This type of structure is now known as a "superlattice."

The theory of the superlattice is closely associated with the theory of the intermetallic compound. While there is no sharp line of distinction between many superlattice structures and intermetallic compounds, two factors generally contribute to determine where two given elements will combine in the form of superlattice structures,

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intermetallic compounds, or ordinary solid solutions. The first of these is known as the "size-factor" which is based upon the closest distance of approach of the atoms within the crystal structure of the solute and the solvent. It has been demonstrated that if the atomic diameters of the solvent and solute differ by more than about 15%, the range of solid solution formation is very restricted, whereas if the difference in atomic diameters is below the limit specified, considerable solid solutions may be formed. The other factor which enters into the problem is the electrochemical factor, by which is meant the degree of separation of the two metals under consideration in the electromotive series. In general, the more electronegative the solute, and the more electropositive the solvent, and vice versa, the greater is the tendency for the formation of stable intermediate compounds.

The formation of a superlattice structure is often accompanied by marked changes in the physical and mechanical properties of the alloy, giving rise to the belief that such superlattice structures may be regarded as definite chemical compounds. However, this cannot be considered the general rule, since from more recent work, it appears that the forces holding the atoms together in a superlattice may be of the same nature as those of a simple metal or a random substitutional solid solution. If these normal metallic linkages are regarded as a type of chemical combination, then the superlattice structures could properly be considered as definite chemical compounds. On the other hand, if the concept of chemical combination is limited to the formation of homopolar or purely ionic compounds, then the superlattice type structures are not, strictly speaking, true chemical compounds.

Because there is no sharp line of demarcation between superlattice structures and intermetallic compounds, the compositions of the present invention will be referred to as intermetallic compounds. The evidence obtained thus far indicates that the compound Ni_3Al has a crystalline structure of ordered face centered cubic lattice. While the size factors are very favorable to the formation of stable solid solutions (nickel has a crystal lattice in which the closest distance of approach of atoms is approximately 2.5 Angstrom units, while in aluminum, the closest distance of approach between atoms is approximately 2.8 Angstrom units) nickel is not separated by a very substantial degree from aluminum in the electromotive series. Consequently, the nature of the forces bonding the atoms together in the ordered structure is not capable of precise definition. Whatever the nature of the forces bonding the elements together, we have found that the compound Ni_3Al has very desirable oxidation resistance characteristics, and hot strength properties not possessed by any other combination of these metals, so far as we are aware.

An object of the present invention is to provide an improved oxidation resistant composition including an intermetallic compound of the type represented by the formula Ni_3Al .

Another object of the present invention is to provide a method for the production of stable, intermetallic compounds produced by diffusing metallic atoms into an intermetallic compound containing aluminum and lower molecular proportions of the metal being diffused therein than is desired in the final intermetallic compound.

Another object of the present invention is to provide a method for promoting solid state diffusion between nickel and intermetallic compounds of nickel and aluminum.

Still another object of the present invention is to provide an improved method for producing a compacted, sintered mass consisting essentially of Ni_3Al .

Formation of the compound Ni_3Al requires special

technique because of the very restricted area of the nickel-aluminum phase diagram in which the compound exists as a stable phase. Merely mixing metallic aluminum and metallic nickel in the proper atomic proportions and then heating the mixture to elevated temperatures does not produce the desired intermetallic compound, because an oxide scum forms on the aluminum causing significant amounts of the aluminum to be unavailable for reaction with nickel. In the process of the present invention, metallic nickel is reacted with the intermetallic compound NiAl under conditions of solid state diffusion, and in the proper stoichiometric proportions to yield the compound Ni₃Al.

The compound NiAl is not essentially oxidation resistant, and is too brittle to be used as a structural material. This compound exists as a body centered cubic crystal lattice and we have found that this structure can be transformed into the ordered face-centered cubic lattice associated with the compound Ni₃Al by a process of solid state diffusion.

The single drawing submitted with this application illustrates a portion of the nickel-aluminum phase diagram in which the compound Ni₃Al exists. As seen from an inspection of this diagram, the area in which the compound Ni₃Al and the liquid exists in equilibrium is very limited. It will be noted from the diagram that the Ni₃Al field extends from the solidus temperature, about 2525° F., down to room temperature, and is adjacent to the field of NiAl plus Ni₃Al on the aluminum side of the diagram and the field of nickel solid solution plus Ni₃Al on the nickel side of the diagram. Since the presence of the brittle compound detracts from the oxidation resistance and workability of the final compositions, it is highly desirable to stay within the field of homogeneous Ni₃Al, or to favor an excess of nickel and form the combination of the nickel solid solution and Ni₃Al, in preference to the combination of NiAl and Ni₃Al.

In essence, the process of the present invention involves mixing the compound NiAl and an amount of additional metallic nickel at least sufficient to yield the compound Ni₃Al, and sintering the resulting mixture at a sintering temperature near but not substantially in excess of the solidus temperature of the compound Ni₃Al. After sufficient time has elapsed to permit solid state diffusion, we have found that the resulting mixture contains the substantially pure compound Ni₃Al to the exclusion of the compound NiAl.

The solid state diffusion reaction can be accelerated if finely divided particles are employed in the initial reaction. Consequently, we prefer to use particles having a particle size on the order of 325 mesh and smaller. Since the compound NiAl is quite brittle, it can be pulverized into an extremely fine state of subdivision. The particulated mixture of NiAl and metallic nickel of the particle size indicated is compacted from a few to 50 tons per square inch, depending on the density to be achieved. The compact is then heated in a high temperature furnace. The proportion of nickel added is substantially equivalent to the stoichiometric amount of nickel required to produce the compound Ni₃Al from the existing NiAl. The resulting composition will therefore have an aluminum concentration of about 13.5% by weight, with the balance being substantially nickel. As previously mentioned, the presence of NiAl in the final product is not desirable, so that in order to insure complete reaction, it is desirable to provide a slight excess of nickel over that stoichiometrically required to produce the compound Ni₃Al. The resulting composition should, however, contain an aluminum concentration of 13.5% by weight, plus or minus 1%, or otherwise the compound Ni₃Al does not result.

In order to further hasten the diffusion of the added nickel into the NiAl, the mixture can be heated above the melting point of nickel initially and then the tem-

perature may be dropped to the sintering range of 2300° F. to 2550° F. For example, the mixture can be heated to a temperature of 2600° to 2650° F. for a period of about 5 to 10 minutes and then the power of the electric furnace turned off until a temperature in the range of 2300° F. to 2550° F. is achieved, whereupon the furnace is maintained at temperatures in the last named range for the desired period of time. Within this broader range, the preferred sintering temperature is in the range of 2300° F. to 2400° F.

The period of time required for complete solid state diffusion of the added nickel into the NiAl to form Ni₃Al is ordinarily on the order of one to three hours, although a period of time from one to 25 hours can be employed, depending upon the temperatures used. To avoid oxidation during heat treatment, the furnace should have a non-oxidizing atmosphere such as hydrogen, argon, neon, helium, or the like.

The process is generally one of diffusion of metallic nickel into the body centered cubic NiAl, resulting in a lattice rearrangement to form an ordered structure of face centered cubic Ni₃Al. This diffusion process may or may not be carried to completion depending upon the time-temperature relationship used.

The resulting compacted, sintered mass consisting essentially of Ni₃Al has improved oxidation resistance characteristics, thus making the compacts highly desirable for use as structural elements in the manufacture of jet turbine engine parts. Tests have indicated that the composition Ni₃Al can withstand oxidizing temperatures of 1600° to 2000° F. without significant loss due to oxidation. Further, the sintered compact can be machined and has a high hot strength, as well as being tough and relatively ductile. All of these advantages clearly distinguish the compositions of the present invention from ordinary alloys of nickel and aluminum which for the most part are not adapted for use in high temperature environments.

While the foregoing discussion has been concerned with the preparation of the compound Ni₃Al, the methods involved are equally applicable to the production of comparable aluminides of cobalt and iron (such as for example Co₃Al and Fe₃Al), with appropriate changes in the sintering temperatures for these metals.

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

We claim as our invention:

1. The method of preparing the intermetallic powdered compound Ni₃Al which comprises mixing the compound NiAl and an amount of additional powdered nickel sufficient to yield the compound Ni₃Al, and compacting and sintering the resulting mixture at a sintering temperature near the solidus temperature of the compound Ni₃Al for a sufficient time to cause solid state diffusion and the formation of Ni₃Al in said mixture.

2. The method of preparing the intermetallic compound Ni₃Al which comprises mixing powdered NiAl and a sufficient amount of additional powdered nickel in proportions sufficient to yield the compound Ni₃Al, and compacting and sintering the resulting mixture in a non-oxidizing atmosphere at a sintering temperature in the range from 2300° to 2550° F. for a sufficient time to cause solid state diffusion and the formation of the compound Ni₃Al in said mixture.

3. The method of preparing the compound Ni₃Al which comprises mixing powdered NiAl and a sufficient amount of additional powdered nickel in substantially stoichiometric portions sufficient to yield the compound Ni₃Al, compacting the resulting mixture, and sintering the resulting compact in a non-oxidizing atmosphere at a sintering temperature in the range from 2300° to 2550° F. for a sufficient time to cause solid state diffusion and the formation of the compound Ni₃Al in said mixture.

4. The method of preparing the intermetallic com-

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pound Ni_3Al which comprises mixing powdered NiAl of a particle size not in excess of about 325 mesh and a sufficient amount of powdered nickel of a particle size not in excess of 325 mesh in substantially stoichiometric proportions to yield the compound Ni_3Al , compacting the resulting mixture, and sintering the resulting compact in a non-oxidizing atmosphere at a sintering temperature in the range from 2300° to 2550° F. for a sufficient time to cause solid state diffusion and the formation of the compound Ni_3Al in said mixture.

5. The method of preparing the compound Ni_3Al which comprises mixing powdered NiAl and a sufficient amount of additional powdered nickel in substantially stoichiometric proportions to yield the compound Ni_3Al , compacting said mixture, heating the resulting compact to a temperature slightly higher than the melting point of nickel, reducing the temperature to a value below the melting point of nickel and in the range from 2300° to 2550° F., and holding said mixture within said range for a period of time sufficient to cause diffusion of nickel into said NiAl and recovering a sintered compact consisting essentially of Ni_3Al .

6. The method of preparing the compound Ni_3Al which comprises mixing powdered NiAl and a sufficient amount of additional powdered nickel in substantially stoichiometric proportions to yield the compound Ni_3Al , compacting the resulting mixture, heating the resulting compact to a temperature in the range from 2600° to 2650° F., reducing the temperature to a value in the range from 2300° to 2550° F., and holding said mixture at a temperature within said last named range for a period of time sufficient to cause diffusion of nickel into said NiAl and recovering a sintered compact consisting essentially of Ni_3Al .

7. The method of preparing the compound Ni_3Al which comprises mixing NiAl and a sufficient amount of ad-

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ditional nickel in proportions sufficient to yield the compound Ni_3Al , compacting the resulting mixture, and sintering the resulting compact in a non-oxidizing atmosphere at a sintering temperature in the range from about 2300° to 2400° F. for a sufficient time to cause solid state diffusion and the formation of Ni_3Al in said compact.

8. The method of preparing the compound Ni_3Al which comprises mixing NiAl and a sufficient amount of additional nickel in proportions sufficient to yield the compound Ni_3Al , compacting the resulting mixture, and sintering the resulting compact in a non-oxidizing atmosphere at a sintering temperature in the range from about 2300° to 2550° F. for a period of one to 25 hours to cause solid state diffusion and the formation of Ni_3Al in said compact.

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