



EFFECT OF ALLOYING ELEMENTS ON PRODUCTION
OF DUPLEX PREFORMS WITH COMPOSITE PROPERTIES

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ABSTRACT

An experimental investigation was conducted to produce cylindrical powder preforms with high hardness at rim and enough toughness at core to suite the requirements in gearing systems. The difference in properties was achieved only by the different alloying additives. A special composite die was designed to perform the partial compaction of either rim or core beside the final compaction of the specimen as a whole. Preforms were produced by 600 MPa compaction pressure and 2 hours sintering under H₂ atmosphere at 1200°C. A series of experiments were carried out using different compositions to achieve the required difference in properties between the core and the rim and secure enough bond and adhesion between them. A mixture consisting of Fe + 5% Cu in the core and Fe + 2% C + 5% Cu + 6% Ni in the rim yielded a compact allowing a shear strength value of 225 N/mm² at the interface, hardness value of 412 HV in the rim, hardness value of 125 HV, in the core, and an overall sintered density of 7.26 gm/cm³. Another series of experiments were carried out using manganese as alloying additive beside copper and nickel to improve the properties at the interface. A mixture consisting Fe + 5% Cu in the core and Fe + 2% C + 2% Cu + 6% Ni + 2% Mn in the rim yielded a compact allowing a shear strength value of 250 N/mm² at the interface, hardness value of 232 HV in the rim, hardness value of 112 HV, in the core and an overall sintered density of 7.28 gm/cm³.

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INTRODUCTION

Attention has been focused on the powder metallurgy techniques to provide certain composite materials with distinct physio-mechanical properties and which can undergo the normal material processing as a mean of production offering components suitable for certain service conditions where the requirements on the surface are different from those on the core(1). Such duplex structure saves material and complicated heat treatments to achieve such requirements(2). Duplex consists mainly of compaction and sintering of two different mixtures of powdered metals or other elements to obtain the final product without further processing. Examples of these mixtures can be prepared to obtain a combination of properties per single component such as hard surface with tough core suitable for production of gearing systems, corrosion resisting outerlayer with sufficiently ductile core, highly conductive skin layer with high strength core, and other similar examples.

The present work shows an investigation to study the production of duplex preforms of composite mixtures and to find the effect of the different alloying elements in these mixtures to achieve from one side, a homogeneous continuous material all over the section with sufficient bond at the interface between an outer rim and an inner core, and from the other side a distinct difference in mechanical properties between this core and the outer rim.

EXPERIMENTAL PROCEDURE

The basic powder used for the production of preforms, and which is considered as the main constituent in either rim or core was the HOGANAS iron powder RZ 150 with average grain diameter $63\mu\text{m}$ and apparent density 2.6 gm/cm^3 . Beside iron, graphite powder KROPFMUHL FP 96/97, carbonyl nickel powder ONIA, and electrolytic copper and manganese powders BAUDIÉRE were used as additives to the different mixtures of both rim and core. Mixtures were prepared by weight from the different elements and mixed together for 5 hours in a ball mill mixer. The composition of the core mixture was fixed to be Fe + 5% Cu while the composition of the rim mixture was varied to be Fe + 2% C + (2% up to 10%) Cu, Fe + 2% C + (0% up to 7%) Cu + (2% up to 8%) Ni, and Fe + 2% C + 2% Cu + (2% up to 6%) Ni + (1% up to 15%) Mn.

The preforms were simply slugs consisting of a rim having the shape of ring with outer diameter $\phi 16\text{ mm}$ and inner diameter $\phi 9\text{ mm}$, at the moment of partial compaction, and a core in the form of solid cylinder $\phi 9\text{ mm}$. The processes of partial compactions and the final compaction were carried out in a specially designed compaction die shown in fig.1. The sequence of compaction was as follows : firstly compacting the ring mixture under the pressure of 200 MPa, then the core mixture is inserted and compacted by the same pressure, at last the final compaction was carried out in the same die under 600 MPa.(3). All specimens were sintered at 1200°C for 2 hours (4) under hydrogen atmosphere in a tube furnace type ADAMEL 50H2 which allows a maximum heating temperature of 1400°C under controlled atmosphere. The rates

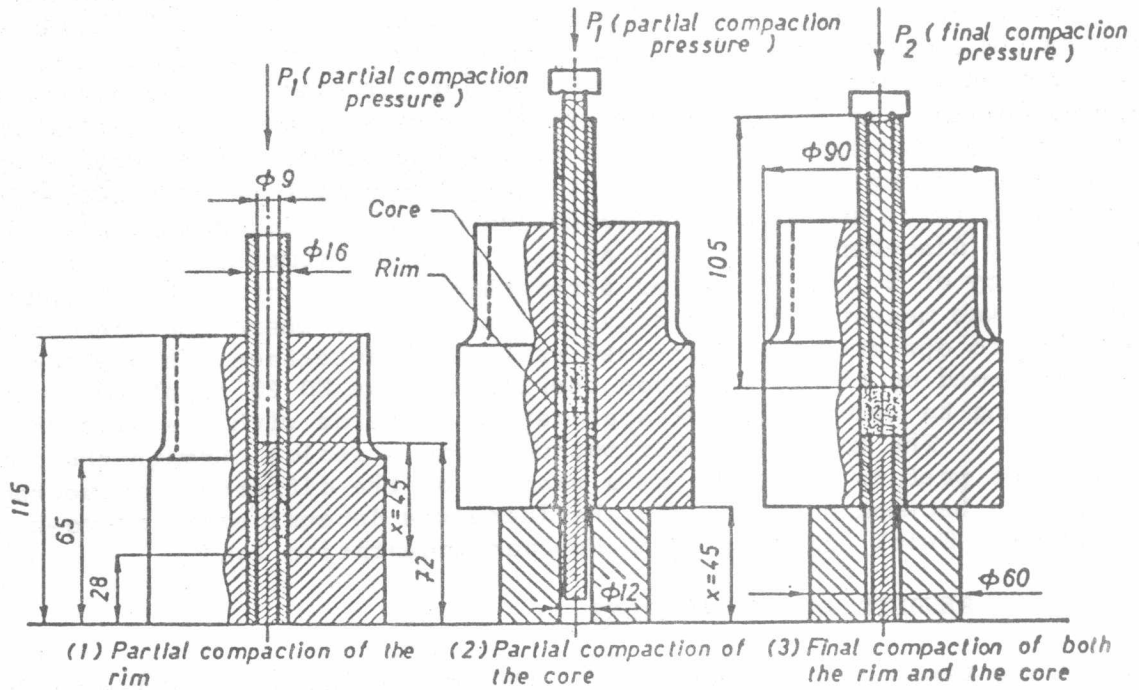


Fig.(1) The sequence of compaction in the designed composite compaction die.



Fig.(2) Microstructure observed at the interface showing the separation between core and rim.



of heating and cooling were in the order of 300°C/hr .

Density of all preforms was measured according to the immersion method. Specimens were first treated with a mixture of acetone + 0.1% silicon oil before the measurements to prevent the ingress of water. Since the control of dimensional change is required in powder preforms and can secure successful duplex, the effect of different alloying elements on either positive or negative dilatation of the matrix was studied. The measurements of dimensional change were carried out along the direction of application of compacting force and along the direction normal to it, namely along the height and diameter of specimen. An electronic micrometer was used allowing an accuracy of $5\ \mu\text{m}$. The shear strength along the interface between rim and core was measured in a specially designed die where the piercing plunger had to operate on the assumed mechanical interface. This shear test was performed on the compression testing machine ZD 20 supplied by spherical bearing to secure coaxial application of force with the axis of the specimen. The hardness distribution along a radius through the cross section of preform was taken as a measure of the difference of mechanical properties between core and rim. Hardness was determined by micro-hardness tester Zwick 3212 using 50 Newtons indentation load.

RESULTS AND DISCUSSIONS

Carbon was taken at the beginning as the only alloying additive to iron powder of the rim to establish the required difference of properties between core and rim. Fig. 2, shows the microstructure obtained at the mechanical interface by the preforms comprising plain iron powder in the core, and Fe + 2% C (5) in the rim, after compaction and sintering. We can distinguish two distinct structures on both sides of the interface, pearlitic in the rim and ferritic in the core. We can remark also a pronounced separation at the interface and on the other hand no common grains cross the interface indicating that separation took place before complete austenitization. This composition achieved the required difference of properties between the core and the rim but it did not secure the bond and complete adhesion between them. This can be explained by the different thermal expansion and contraction characteristics obtained during sintering by the core powder and the rim mixture.

Attempts to overcome this problem made it necessary to add elements of swelling effect to the core mixtures and elements of shrinkage effect to those of rim (6,7,8,9). Different percentages of copper were added to the rim mixture while fixing the percentage of copper in the core mixture to be 5%. Fig. 3, shows the effect of copper additives to the rim mixture on the sintered density of preforms. It can be noticed that the density increases with increasing the copper content up to 5% and then it drops very sharply at higher percentages of copper and even it may attain lower values than that measured without addition of copper in the case of rim mixture containing 10% Cu. This behaviour can be explained by the fact that copper in such mixture, and at the adopted temperature of sintering forms liquid phase which is from one side favourable for the processes of diffusion, bridging, and welding of iron particles thus increasing the density, and from another side, copper by the high rate of liquid diffusion allows the formation of solid solution and eutectoid structure with iron which causes an increase in the volume (10). The amount of this

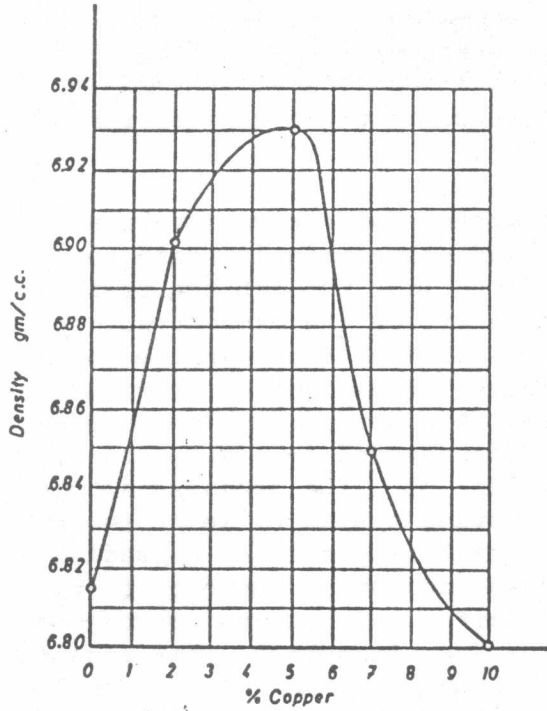


Fig.(3) Effect of Copper percentage in the rim mixture with fixed percentages in the core on the sintered density of preforms.

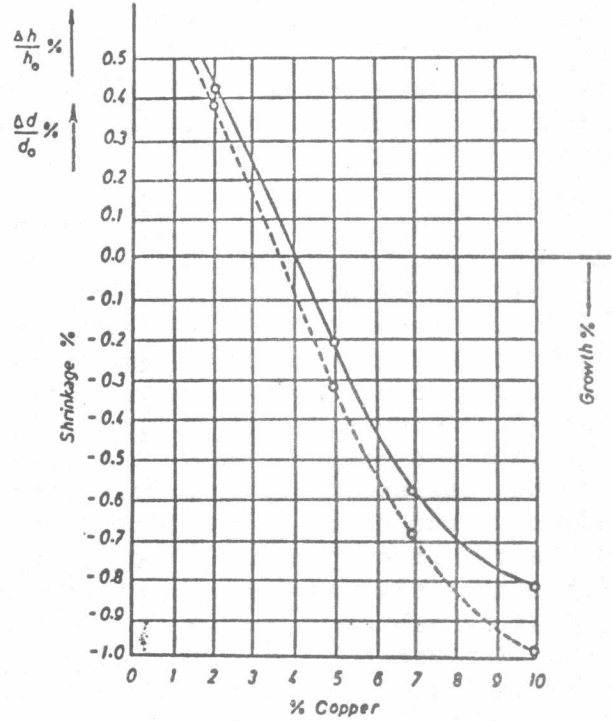


Fig.(4) Effect of Copper percentage in the rim mixture with fixed percentages in the core on the linear dimensional change of preforms.



Fig.(5a) Ferritic-Pearlitic structure observed at both sides of the interface.

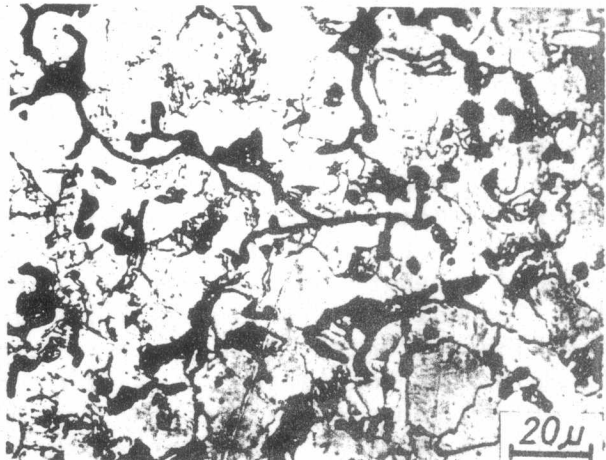


Fig.(5b) Ferritic structure with iron-copper eutectoid on the grain boundaries observed at the centre of specimen.

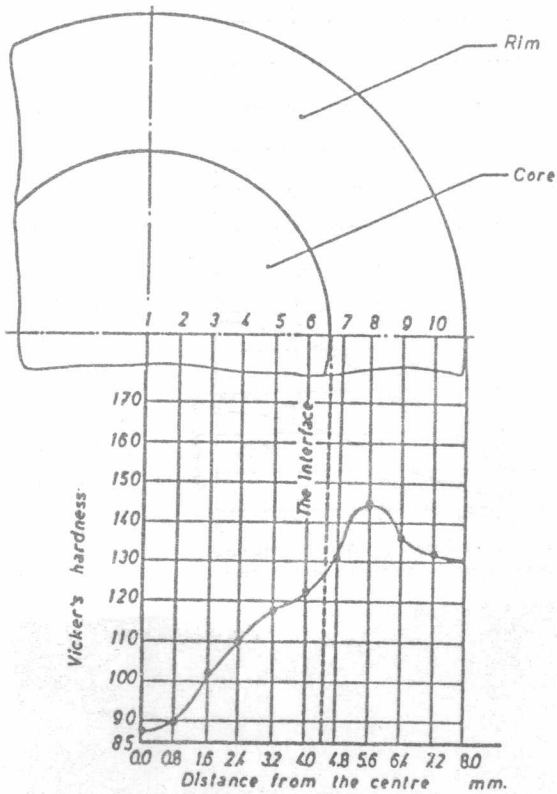


increase in volume is more pronounced at percentage higher than 5% Cu, which is the origin of the recorded decrease in density. The dimensional changes measured for the preforms with different percentages of copper additions are shown in Fig.4. We can observe a clear swelling effect in the preforms with percentages higher than 4% Cu. Swelling is considered as a major requirement in the core in order to eliminate the microgap between rim and core.

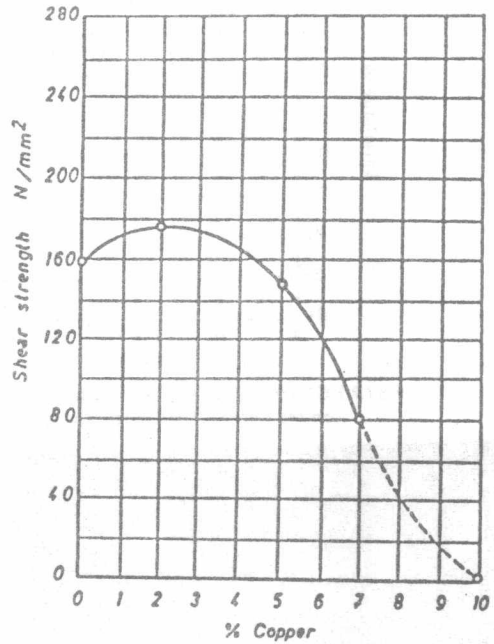
The microstructure observation proved the absence of separation between rim and core, however due to the existence of copper liquid phase the diffusion of carbon became easier in all directions through the preform and it was seen that in both sides of the interface the microstructures manifest themselves in the form of pearlite combined with ferrite fig.5a. In the direction of the centre of the specimen the amount of ferrite is gradually increasing at the expense of pearlitic structure. Iron-copper eutectoid was observed along the grain boundaries and specially pronounced when the structure is mostly ferritic fig. 5b. The hardness along a radius of the specimen is shown in fig.6, having a peak value of 145 HV in the rim corresponding to microstructure rich in pearlite. A gradient of hardness towards the edge of the specimen indicating the fact that partial decarburization took place on the surface of preform due to the carbon interaction with the hydrogen protecting atmosphere. This results in migration of some carbone atoms to the outer direction to replace the lost amount on the surface. The results of the shear test applied to preforms having different contents of copper in the rim, keeping the core with 5% Cu, are shown in fig.7. Shrinkage of rim with only 2% Cu, and swelling of core by the effect of 5% Cu, gave a highest shear strength value of about 175 N/mm². Increasing the percentage of copper over 7%Cu in the rim separation appears again.

Reaching this state at solution of the problem of duplex préforms it was possible to eliminate the separation by adequate amounts of copper in both core and rim mixtures. But as it was clearly seen, through the microstructure investigation, that the carbon found its way to the core due to the diffusion phenomenon enhanced by the liquid phase sintering. To achieve a property difference between core and rim, carbon trap should be arranged to keep it mostly in the rim. Nickel was chosen to be added to the rim mixture beside carbon and copper to obtain the benifit of its double action (11). Firstly nickel has a contraction effect that may improve the bond strength between rim and core when it is added to the rim mixture. Secondly, nickel is a strong austenitic forming element that may stabilize austenite even to room temperature, phase structure which allows higher solubility of carbon. From the other side, the presence of nickel may enhance the formation of higher strength structure phases. Nickel was added in different amounts up to 8% combined with different percentages of copper to the rim mixture while keeping the mixture of core constant (Fe + 5% Cu).

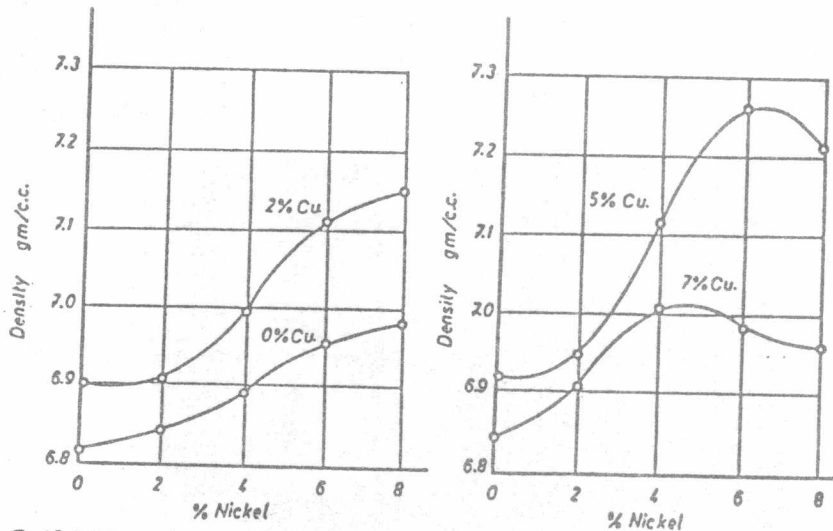
Densities of preforms with different contents of nickel are shown in fig.8, which indicates that a maximum density could be obtained by a mixture of 5% Cu and 6% Ni in the rim. The dimensional variations of preforms in pressing and lateral directions as a function of copper and nickel contents in the rim mixture are illustrated in fig.9, where we can realize that by increasing the copper content the shrinkage values



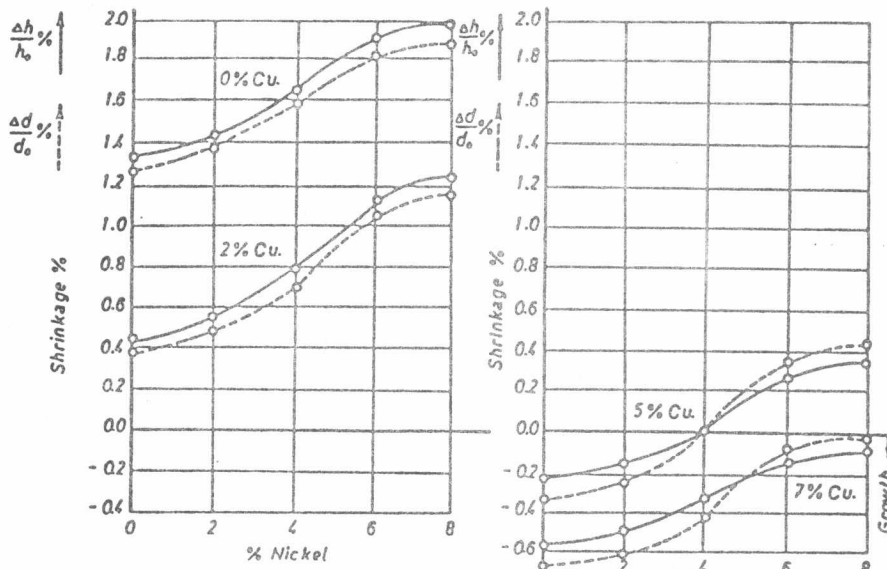
Fig(6) The Vicker's hardness distribution along a radius of the preform having Fe+2%Graphite+5%Cu for the rim mixture and Fe+5%Cu for the core mixture.



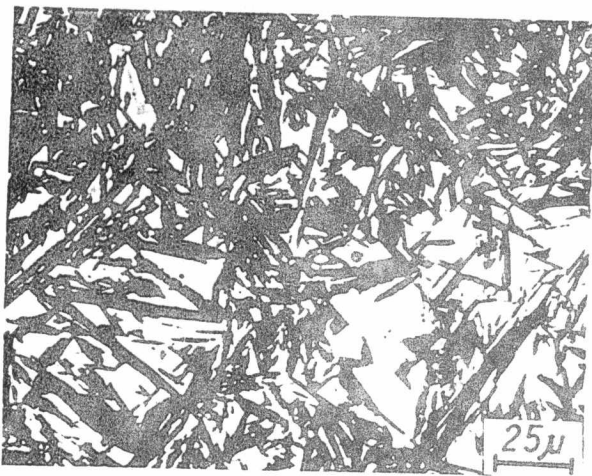
Fig(7) Effect of Copper percentage in the rim mixture with fixed percentages in the core on the shear strength at the interface.



Fig(8) Effect of Nickel percentage for various percentages of Copper in the rim mixture on the sintered density of preforms having fixed percentages in the core.



Fig(9) Effect of Nickel percentage for various percentages of Copper in the rim mixture on the linear dimensional change of preforms having fixed percentages in the core.



Fig(10a) Microstructure observed at 1.45 mm from the interface towards the edge of specimen.

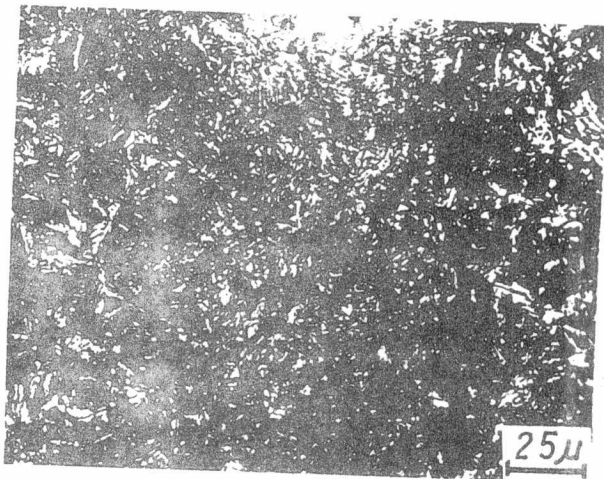


Fig.(10b) Microstructure observed at 0.45 mm from the interface towards the edge of specimen.

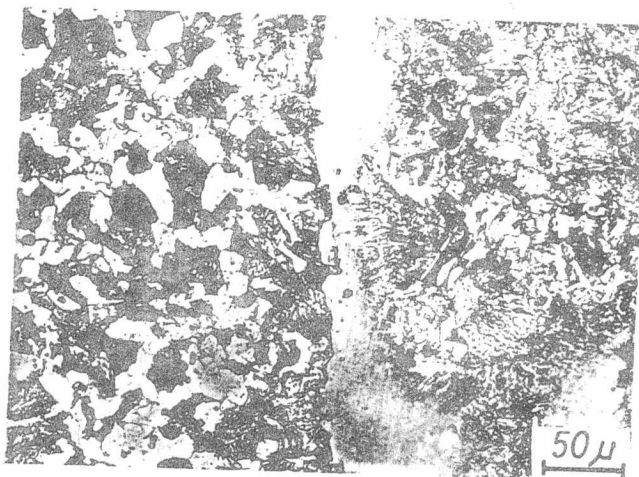


Fig.(10c) Microstructure observed at the interface between rim and core.

are decreased and even inverted to negative values (swelling) as seen in the case of the mixture containing 7% Cu, in agreement with the behaviour of the density curves. From the other hand the mixture containing 5% Cu and 4% Ni represents a critical mixture since zero dimensional change can be achieved by this composition. This proves that by the control of alloying additions in powder products we can control dimensional changes after sintering (12). As a result of the study of both density and dimensional changes, the mixture which comprises beside iron and carbon, 5% Cu and 6% Ni was selected since it provides a reasonable positive shrinkage of the rim and in the mean time achieves the highest possible density of preforms. The microscopic examination showed that the rim starting from the edge of specimen till the interface comprises a mixture of austenite inside which colonies of martensite exist mixed with lower bainite structure. This complex structure continued till the interface where a barrier of austenite was formed just adjacent to the interface with the core. This barrier may explain the capturing of carbon in the rim which assisted the formation of martensitic and bainitic structures. In the core just beside the interface the structure is remarked by rich amount of pearlite that explains early diffusion of carbon during the sintering operation. Towards the centre of the core the amount of ferrite increases at the expense of pearlitic structure. The micrographs figs. 10a, b, c, d, e, illustrate the different microstructures along a radius within a specimen containing Fe + 5% Cu, in the core and Fe + 2% C + 5% Cu + 6% Ni, in the rim.

The hardness values measured along a radius of the above specimen are shown in fig. 11, where a peak value of 412 HV is obtained in the rim with a rapid decrease in hardness towards the interface followed by a smooth gradient through the core to the centre of specimen. The decarburisation phenomenon is manifested by a decrease of the value of hardness on the surface. The shear strength was measured on specimens having different percentages of copper and nickel. The values of shear strength are shown in fig. 12. Generally by the increase of nickel percentage the shear strength is increased. The highest values of shear strength were achieved by the group of specimens having 5% Cu. The highest value is 245 N/mm^2 reached at 8% Ni and reasonable value of 225 N/mm^2 reached at 6% Ni. These values of shear strength introduce the chance to compare between the two groups of 6% Ni, from one side, and 8% Ni from the other side, fixing the copper content to be 5% Cu. A hardness test was carried out along a radius of specimen with 8% Ni and the measured values are shown in the same fig. 11. A surprising phenomenon appeared due to the effect of nickel higher than 6% in these mixtures, where extremely high rate of carbon diffusion across the interface causes accumulation of carbon in the core instead of the rim. Such effect results in a peak of hardness equal to 232 HV in the core. Therefore, in spite of the higher shear strength values obtained by the mixture with 8% Ni, the mixture with 6% Ni is preferable since it allows the achievement of a duplex preform with the required difference in properties between rim and core beside having the advantage of obtaining the highest density and a reasonable shears strength.

Beside copper and nickel, manganese was added to the rim mixture to study its effect on the final properties of preforms while keeping the core mixture to be Fe + 5% Cu. Since manganese has a swelling effect (13,14), then the amount of copper is reduced down to 2% Cu. A series of specimens with different manganese content for various percentages of nickel were examined and the resulting densities of preforms are shown in fig. 13. A

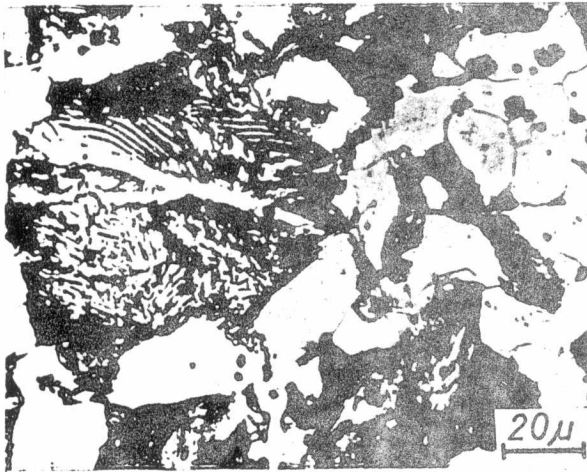


Fig.(10d) Microstructure observed at 1 mm from the interface towards the centre of specimen.

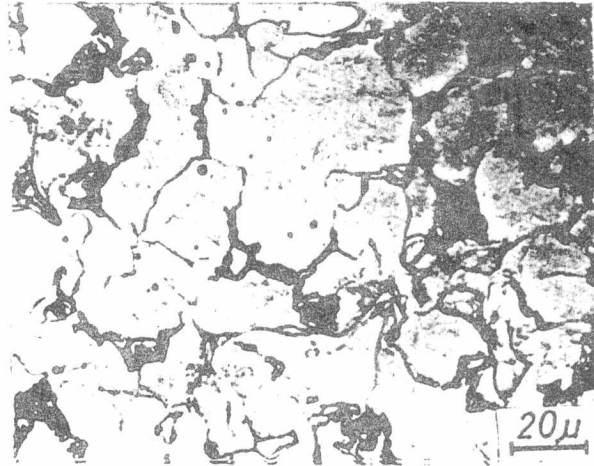
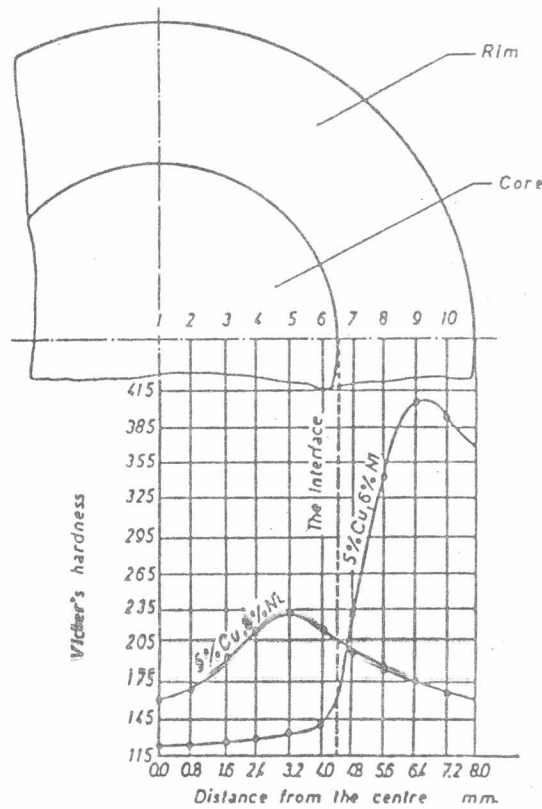


Fig.(10e) Microstructure observed at the centre of specimen.



Fig(11) The Vicker's hardness distribution along a radius of the preforms having Fe+2% Graphite+5%Cu+(6%Ni, 8%Ni) for the rim mixture and Fe+5%Cu for the core mixture.

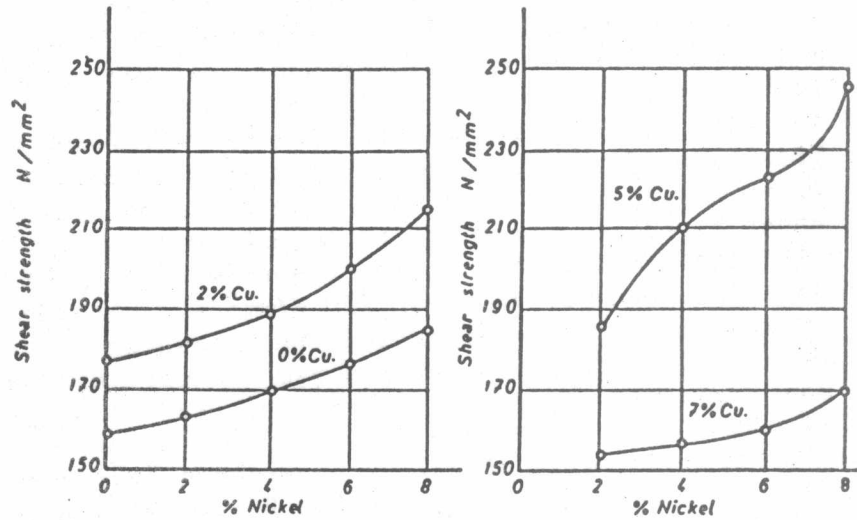


Fig.(12) Effect of Nickel percentage for various percentages of Copper in the rim mixture on the shear strength of preforms having fixed percentages in the core.

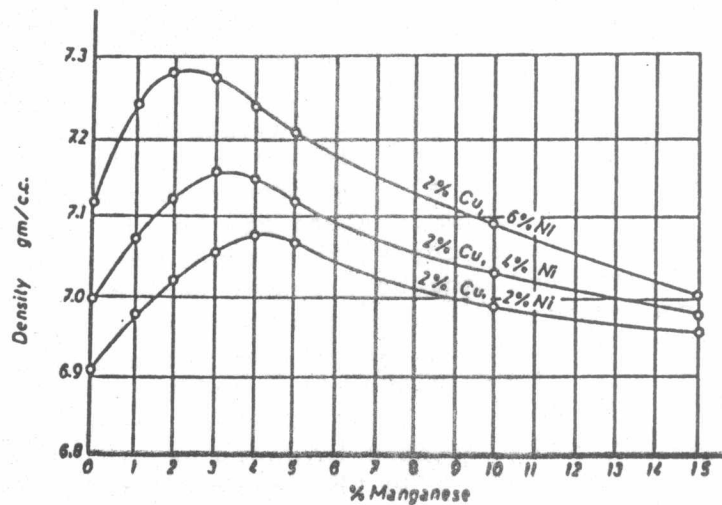


Fig.(13) Effect of Manganese percentage for various percentages of Nickel in the rim mixture on the sintered density of preforms having fixed percentages in the core.

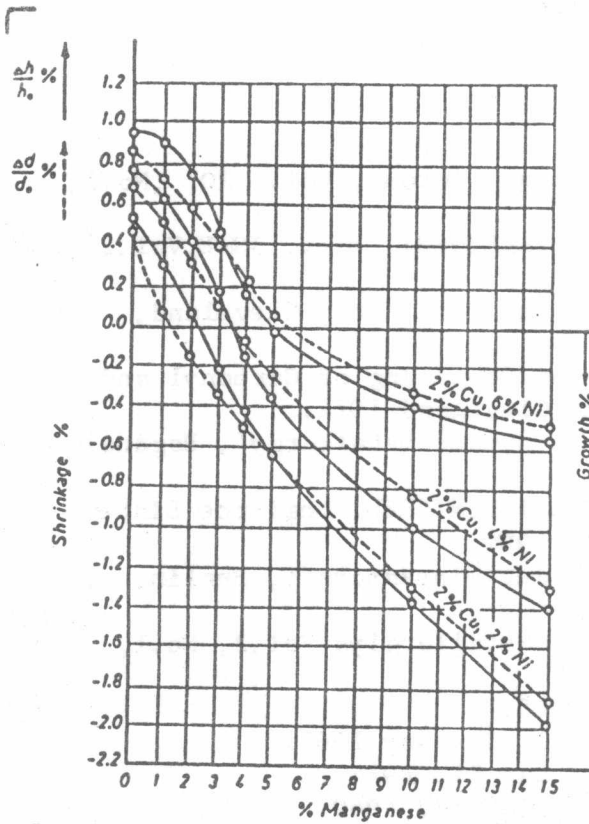


general character is that the density increases by the increase of manganese content till a certain maximum value then a drop in the values of density followed. This fact is due to the interactions amongst the elements present in the mixture, regarding their mutual solubilities or their response for the formation of phases or compounds that also impose their effects on the dimensional change. A highest density value of 7.28 gm/cm^3 was reached in case of the mixture containing Fe + 2% C + 2% Cu + 6% Ni + 2% Mn, in the rim. This same mixture secures a positive shrinkage as indicated by fig. 14, which may increase the bond between rim and core and consequently the shear strength values at the interface. The results of the shear test elaborated on the same group of specimens fig. 15, indicate that a maximum shear strength value of 250 N/mm^2 was obtained by the same mixture. It is also clear that, increasing the manganese content the shear strength decreases and separation between rim and core appears for higher contents of manganese over 5% Mn. The hardness test elaborated along a radius of a specimen of the previous composition is shown in fig. 16, where a maximum hardness value of 232 HV covering a flat peak was reached in the rim and a hardness shelf of about 195 HV is obtained at the interface region, then a hardness gradient through the core is visible.

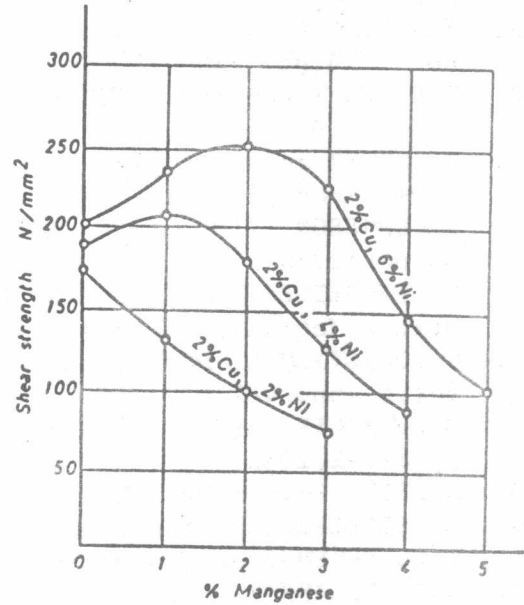
Comparing the bond strength at the interface and the hardness values in the rim obtained by the different systems with different alloying additions we can note that the mixture containing Fe + 2% C + 2% Cu + 6% Ni + 2% Mn in the rim, achieved the highest shear strength value at the interface while the mixture containing Fe + 2% C + 5% Cu + 6% Ni, achieved the highest hardness in the rim.

CONCLUSION

Powder metallurgy technique offer the possibility of Producing composite preforms suitable for gear production where hard surface and simultaneously tough core are required. Such duplex structure can be obtained by compaction and sintering of two different mixtures with different alloying elements to achieve the required properties in both rim and core. The addition of carbon as the only alloying element to the rim iron powder achieved the required difference of properties between core and rim but it did not secure the bond between them. The addition of copper to the core mixture and a proper combination of copper, nickel and manganese beside carbon to the rim mixture yield a preform with continuous material on both sides of the interface and with distinct difference in mechanical properties between rim and core. A mixture consisting of Fe + 5% Cu in the core, and Fe + 2% C + 5% Cu + 6% Ni in the rim, yielded a compact allowing a shear strength value of 225 N/mm^2 at the interface, and a maximum hardness of 412 HV in the rim. Another mixture consisting Fe + 5% Cu in the core, and Fe + 2% C + 2% Cu + 6% Ni + 2% Mn in the rim yielded a compact allowing a shear strength value of 250 N/mm^2 at the interface and a maximum hardness of 232 HV in the rim.



Fig(14) Effect of Manganese percentage for various percentages of Nickel in the rim mixture on the linear dimensional change of preforms having fixed percentages in the core.



Fig(15) Effect of Manganese percentage for various percentages of Nickel in the rim mixture on the shear strength of preforms having fixed percentages in the core.

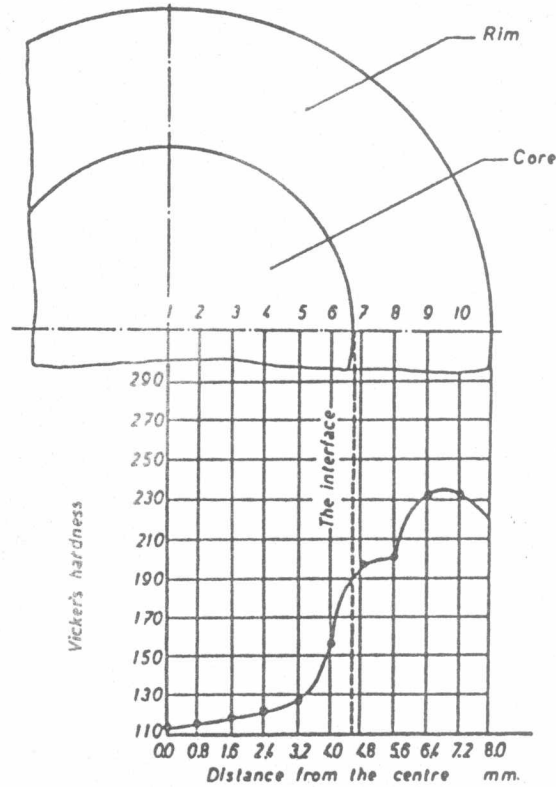


Fig.(16) The Vicker's hardness distribution along a radius of the preform having Fe+2% Graphite+2% Cu+6% Ni+2% Mn for the rim mixture and Fe+5%Cu for the core mixture.



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