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ENHANCED DENSIFICATION OF WHITE CAST IRON POWDERS BY CYCLIC PHASE TRANSFORMATIONS UNDER STRESS

Oscar A. Ruano, Jeffrey Wadsworth and Oleg D. Sherby

ABSTRACT

It is shown that densification of white cast iron powders under stress can be enhanced by multiple phase transformations through thermal cycling. This enhancement occurs by accelerated creep flow during phase changes (transformation superplasticity). The stress range where transformationassisted densification can occur is shown to be between 1.7 MPa (250 psi) to 34.5 MPa (5000 psi). Below 1.7 MPa insufficient strain occurs during phase transformation to cause significant densification even after many transformation cycles. Above 34.5 MPa, densification occurs principally by normal slip creep. Transformation warm pressing of white cast iron powders leads to dense compacts at low pressures and short times. In addition, because the transformation temperature is low, the ultrafine structures existing in the original powders are retained in the densified compacts.

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INTRODUCTION

The advent of new technologies centered on rapidly solidified powders often require development of methods of enhancing densification wherein the fine structures present in such powders are retained. To achieve this goal it is necessary to use low temperatures, but this usually requires the application of high pressures if a high density is to be achieved. High pressures are often a limiting factor in the manufacture of powder products. Methods which permit the reduction of pressure needed for high densification are therefore desirable. One such method of enhancing densification of powders is by accelerating plastic flow through the generation of internal stresses during warm pressing. A technique for generating internal stress is through the use of multiple, solidstate phase transformations (transformation superplasticity) [1-5].

A number of investigations have utilized multiple phase transformations as a possible means of enhancing sintering of powders [6-8]. Little or no significant enhancement in densification was reported in these cases where no applied stresses were used. Kohara [9] extended this work by investigating the densification of iron powders through transformation cycling under extremely small applied stresses (\sim 0.1 MPa (10 psi)). The enhancement of densification observed by Kohara, although limited, was attributed to the occurrence of transformation superplasticity. As will be shown in the next section, however, transformation superplasticity cannot contribute to significant densification under such low externally applied stresses in ferrous base materials. In a separate investigation, Oshida [10] examined the densification of cast iron powders by multiple thermal cycling through the A₁ transformation temperature (727°C) under small applied stresses (0.5 to 1.5 MPa (70 to 210 psi)), and he also attributed the enhancement of densification to transformation superplasticity. The densification

observed, however, cannot be attributed to transformation superplasticity. As will be shown later, under the transformation cycling conditions used by Oshida, significant densification would only be expected if stresses much above 1.5 MPa had been applied. The densification results observed by him will be discussed in the next section.

The above discussion would indicate that the influence of transformation superplasticity on densification of powders has not been resolved. It is the purpose of this paper to examine this area with two objectives in mind. The first is to investigate the possibility of enhancing the densification, by multiple phase transformations, of 2.4% C white cast iron powders manufactured by rapid solidification rate processing. The object in this case is to establish the influence of transformation superplasticity over a wide range of externally applied stresses (3 to 70 MPa (450 to 10,000 psi)). The second objective is to investigate the conditions of temperature and numbers of transformation cycles which will permit retention of the fine structure that is present in the rapidly solidified white cast iron powders.

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In order to understand the principles pertinent to this subject, it is first necessary to evaluate the factors influencing transformation superplasticity. These factors will be described in the next section which will be followed by the specific experimental study chosen for investigation.

PRINCIPLES OF TRANSFORMATION SUPERPLASTICITY APPLIED TO POWDER DENSIFICATION

The internal generation of stresses during plastic flow may result in high strain rate sensitivities and low strengths which in turn can lead to superplastic behavior [1-5]. One method of obtaining superplasticity of this type is through repetitive, solid-state phase transformations under the simultaneous presence of an externally applied stress. This is because during the phase transformations, volume changes occur which create internal stresses and these stresses aid plastic flow. Studies have revealed that the strain produced from phase transformations is proportional to the applied stress [11-13]. Furthermore, at a given applied stress, σ , the plastic strain which results from a phase transformation increases with 1) an increase in the volume change occurring during transformation, and, 2) a decrease in the strength of the phases involved [14,15]. In equation form, the following relation can be written

$$\varepsilon_{\alpha \neq \beta} = C(\frac{\Delta V}{V}) \qquad \frac{\sigma}{\sigma_{\alpha \neq \beta}} \tag{1}$$

where $\varepsilon_{\alpha \neq \beta}$ is the strain from a two-way transformation, $(\Delta V/V)_{\alpha \neq \beta}$ is the relative change in volume from α to β , $\sigma_{\alpha \neq \beta}$ is the strength or hardness of the weakest of the two phases involved, and C is a constant. The validity of Eq. (1) for a number of polymorphic metals is shown in Fig. 1 [14-18]. Since the rate of heating and cooling through the transformation is usually about the same in most investigations (about 5°C per min), one can consider that the time during transformation, when the strain $\varepsilon_{\alpha \neq \beta}$ occurs, is a constant. Thus, the relation between strain rate, $\dot{\varepsilon}_{\alpha \neq \beta}$, and the applied stress, σ , is also represented in Fig. 1. The relation is observed to be about linear at low stresses. Therefore, the strain rate sensitivity exponent, m, is unity in the equation relating flow

stress, σ , to strain rate, $\hat{\epsilon}$ ($\sigma = K \hat{\epsilon}^{\mathbf{m}}$, where K is a constant). This is an important characteristic because high strain rate sensitivities usually lead to superplasticity [1-5,19]. In addition to exhibiting high tensile ductility, such materials are also very weak and deform at stresses where little or no normal creep would be expected.

There have been a number of investigations on the influence of stress on the transformation strain of ferrous base materials [11-13]. Figure 2 summarizes data on these materials plotted as strain during two-way transformation versus stress. A number of variables need to be considered to explain the spread in the data. Curves 1 and 2 in Fig. 2 are for iron undergoing transformation at about 910°C. The large transformation strain noted here can be attributed to the large volume change $(\frac{\Delta V}{V})$ $\alpha \neq \gamma$ during transformation, and to the low strength of the unalloyed iron, $\sigma_{\alpha \neq \gamma}$, compared to the other ferrous materials. Curves 3, 4 and 5 are for iron containing about 0.2% carbon. For each material, transformation occurs in the temperature range of 727°C (the A_1 temperature) to about 850°C (the A_2 temperature). The difference in transformation strains observed for the three Fe-C alloys can be attributed to the influence of alloying effects and to heating rate effects on transformation superplasticity. Thus, the addition of 0.46% Mn (curve 4), which is seen to decrease the transformation strain of Fe-0.2% C (curve 3), is probably due to The threefold decrease in the influence of manganese on increasing $\sigma_{\alpha \neq \gamma}$. transformation strain observed in curve 5, compared to curve 4 is probably due to an increase in the rate of transformation cycling. Curve 4 was obtained from tests where transformation cycling was carried out at heating-and-cooling rates of 2°C per min, whereas curve 5 was obtained from tests where transformation cvcling was carried out at heating-and-cooling rates of 250°C per min. This is in agreement with the observations made on unalloved iron where it

was shown that very rapid rates of transformation cycling can lead to insignificant transformation strains [12]. Curve 6 in Fig. 2 is for a 1% C steel (52100) transformed at a high heating-and-cooling rate of 250°C per min. Transformation for this material occurs between 727°C and about 750°C. The low transformation strain observed for this material can thus be attributed principally to the high heating-and-cooling rate used.

The above discussion permits the establishment of upper-and-lower bounds of stress where the enhancement of densification can be expected for ferrous powders that exhibit transformations in the range 727 to 800°C (e.g. white cast iron powders). These bounds are shown by the dashed vertical lines given in Fig. 2 and are based on transformation cycling at heating-and-cooling rates in the order of 5°C per min. The lower bound is shown at about 2 MPa. At stresses below 2 MPa no significant densification by transformation superplasticity should occur. This is because the strain for a single two-way transformation is only ϵ \approx .002 (see curves 3 and 4 in Fig. 2). Such a transformation strain, even if repeated fifty times yields a total strain of $\varepsilon = 0.10$ which would not lead to near-full densification. This is based on the assumption that a true strain of at least $\varepsilon = 0.2$ is required to cause full densification. Performing more transformations at this heating-and-cooling rate is considered to be technologically impractical because too long a time would be needed to achieve full densification. Increasing the transformation cycling rate is only of limited assistance in densification since the transformation strain is reduced as the heating and cooling rate is increased

as discussed above^{*}. The upper bound for densification through transformation superplasticity is shown as 30 MPa in Fig. 2. Above this stress, normal creep processes begin to take on a dominant role and will overshadow the contribution from transformation superplasticity. For example, at 50 MPa, a strain of about 2% is produced by one transformation cycle (Fig. 2). One transformation cycle is accomplished in eight minutes for the heating-and-cooling rate used in this investigation. For comparison, the creep rate of fine-grained white cast iron is about 3% per min at 727°C, at the same stress of 50 MPa [20]. Thus, the normal creep contribution during the heating-and-cooling cycle would be considerably more than that by transformation cycling.

* The lower bound of 2 MPa determined here for practical densification rejects the claim of Oshida that densification is enhanced by transformation superplasticity at stresses between 0.5 and 1.5 MPa [10]. In addition, the very high heating-and-cooling rate used by him (800°C per min) would further diminish the benefit from transformation superplasticity. One must conclude that the densification observed by Oshida is due to other factors. One possible explanation lies in the heating method utilized (direct current heating) which could have led to locally high temperatures at particle interfaces producing normal sintering effects.

MATERIAL AND EXPERIMENTAL PROCEDURE

White cast iron powders were used for the present investigation. The chemical composition of the material was by wt %: 2.36 C, 0.92 Mn, 0.014 P, 0.14 Si, 0.0145 S, 0.018 Cr, balance Fe. These powders have a very fine microstructure [21] as a result of preparation by the rapid solidification rate (RSR) processing developed by Pratt and Whitney [22].

The particle size was measured using a Tyler mesh analysis. Microscopic examination of powders was carried out with both the scanning electron microscope (SEM) and the optical microscope. Samples were etched in 5% nital prior to examination. Optical microscopy of unetched samples was also performed in order to determine porosity at different applied pressures. An areal analysis was carried out from optical photomicrographs to determine the volume porosity.

Samples for densification studies were prepared in the following manner. The 2.4% C white cast iron powders were spread in a thin layer of about 120 µm thickness, between two, flat, mild steel plates that were degreased after surface grinding to remove the oxide. An example of a mild steel/cast iron powder/mild steel assembly is shown in Fig. 3. This method of sample preparation is a unique approach to studying the warm pressing of powders avoiding the problems of frictional stresses that normally arise between the powders and die walls.

An example of the microstructure of the powders densified using the above technique is shown in Fig. 4. Three regions are observed in this figure. At the bottom, a fully dense region of very fine-structured white cast iron is evident. The structure is one consisting of fine particles of

cementite in a ferrite matrix. Adjacent to this region is an interdiffusion zone due to carbon diffusion into mild steel from the white cast iron at temperatures above the A_1 during transformation cycling. This region contains coarse pearlite. The final region, at the top of the photomicrograph, shows the coarse-grained mild steel region.

Warm pressing was performed in a resistance furnace attached to a 22,700 kg capacity, servo-hydraulic, MTS-testing machine programmed to deliver constant load. Temperature was controlled to \pm 3°C and measured using two chromel-alumel thermocouples located at the top and bottom of the sample. An atmosphere of forming gas, 90% N₂-10% H₂, was used to minimize oxidation.

Transformation cycling tests were performed by varying the temperature from 50°C above to 50°C below the A_1 transformation temperature (727°C). The time for a complete cycle (675°C to 775°C and back to 675°C) was about 8 min.

RESULTS AND DISCUSSION

The particle size distribution of as-quenched powders is given in Table I. The average size of particles was 45 μ m. The particles in general had a spheroid shape although some showed irregular forms probably due to collisions between particles during the powder processing.

The as-quenched microstructure of the particles is a complex one consisting of retained austenite, carbides and some martensite [21]. This microstructure changes drastically after annealing for short times (15 min) at 650°C. No further significant changes were observed after much longer annealing times at 650°C. A typical SEM micrograph showing the annealed micro-

TABLE I: Screen Analysis for 2.4% C White Cast Iron Powders

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Tyler Screen Mesh Size, µm	< 45	45	64	106	150	180
% of Charge Weight Trapped	39	24	28	8	1	0

structure of part of a single particle is presented in Fig. 5. In this case, the powder particle has been heated at 650° C for 15 min. The photomicrograph reveals a fine mixture of cementite (light area) and ferrite (dark area)^{*}.

After warm pressing, optical photomicrographs were taken of the pressed powders and these were used for porosity determinations. Micrographs of unetched samples were used in order to accentuate the contrast between the pores and the cast iron powders. Typical micrographs are shown in Fig. 6 for powders that were warm pressed under transformation cvcling conditions using three different applied stresses. As can be seen, for a given number of transformation cycles (in this case one two-way cycle), the porosity decreases with increasing applied stress. For example, at 1.6 MPa, very little bonding between powder particles is observed as shown in Fig. 6A. In this case, it is important to recognize that in addition to porosity there are large empty regions due to the loss of powder particles. This loss most probably occurs during the polishing of the samples and reflects the poor bonding at low applied stress. For this reason, densities below about 80% could not be accurately measured. After one two-way transformation cycle, under an applied stress of 15.7 MPa, the powder particles are fully interconnected, Fig. 6B. Lastly, after one two-way transformation cycle, under an applied stress of 31.3 MPa, a material close to full densification (98.3%) is obtained and the residual pores are small and isolated (Fig. 6C).

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The volume fraction of carbide as measured from the photomicrograph is larger than the equilibrium volume fraction (35%). This is an artifact which arises from etching. Ferrite is preferentially etched and the cementite is brought out in relief leading to an apparently large volume fraction of cementite.

The white cast iron 2.4% C powders were warm pressed over a range of applied stresses (3 to 70 MPa) using a number of different thermal-pressure histories. The five routes used are summarized in Table II. In route 1, a single transformation cycle, under various applied stresses was used. The samples were loaded prior to heating and the pressure was maintained for the entire test. In route 2, ten transformation cycles were given for each of a number of applied stresses. The approximate total times that the samples were held at temperature, during a full transformation cycle, were 8 min for route 1 and 80 min for route 2. The influence of time at 650°C under pressure was investigated by holding samples at this temperature for 1/2 hr (route 3) and 2 hr (route 4) under a range of applied stresses. Finally, in route 5, samples were heated to 775°C, without applied stress, and then the pressure was applied for 1/2 hr and removed prior to cooling. Thus, the influence of pressure on densification at 775°C was evaluated without the contribution of a transformationinduced strain.

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The results of the warm-pressing studies are shown in Figs. 7 and 8. In Fig. 7 the influence of stress on densification at 650°C for routes 3 and 4 is shown. As can be seen, at the highest stress used (69 MPa), full densification was observed at 650°C after times of 1/2 hr and 2 hr. At pressures lower than 69 MPa, the influence of time at temperature is apparent, i.e. the increase of time at a given applied stress leads to an increase in density. At an applied pressure of 20 MPa, for example, less than 80% densification is observed after 1/2 hr at 650°C whereas over 90% densification is observed after 2 hr at 650°C. In Fig. 8 these results are compared with those obtained by transformation cycling while under an applied stress. Once again, the density is found to increase with an increase in applied stress. The results also demonstrate that the influence of transformation cycling during the application of an externally applied stress is a major factor in enhancing densification. For example,

Densification Studies of White Cast Iron Powders Using Five Different Routes TABLE 11:

	Thermal History	Number of cycles under stress	Pressures used, MPa, and densities achieved (%)
ROUTE 1	675°C \$ 775°C	1 (8min)	3.1(<80), 6.3(89.8), 15.7(94.0), 21.9(94.2), 31.3(98.3).
ROUTE 2	675°C 775°C	10 (80 min)	1.6(<80), 3.1(91.6), 6.3(94.8), 15.7(98.1), 21.9(98.6) 31.3(100).
ROUTE 3	650°C, 1/2 h	O	6.9(<80), 15.7(81.6), 31.3(90.7), 48.3(97.2), 68.9(100) .
ROUTE 4	650°C, 2 h	o	6.9(<80), 13.8(90.4), 34.5(96.4), 48.3(98.9), 68.9(100) .
KOUTE 5	775°C, 1/2 h	Ð	6.9(<80), 15.7(81.8), 21.9(90.5), 31.3(97.7).

under a very low externally applied stress of 6.9 MPa, a density of over 95% is found for ten cycles and a density of 90% is found for one cycle. By contrast, densities of much less than 80% are found for the warm-pressed samples at both 650°C and 775°C. Transformation cycling is also seen to enhance the densification of white cast iron powders at high applied stresses. For example, at 20 MPa, 99% densification is found for ten cycles and 95% densification is found for one cycle. Without transformation cycling, only 90% densification is found after warm pressing for 1/2 hr at 775°C and less than 80% densification is found after warm pressing for 1/2 hr at 650°C.

The results shown in Fig. 8 indicate that high densification can be achieved in a short time by utilizing transformation cycling under small applied stresses. As an example, a 98% dense product is obtained in only 8 min when transformation cycled once under an applied stress of 31 MPa (4.5 ksi). This is to be compared with only 90% densification after 30 min at 650°C, under the same stress, when no transformation cycling is performed. These results suggest that transformation cycling may have practical utility in the manufacture of powder metallurgy products because of the short times, low temperatures, and low stresses needed to achieve full densification. Furthermore, under these conditions of powder densification, fine starting structures in the original powders are retained. This is shown in the SEM photomicrographs given in Fig. 9. Figs. 9A and 9B show the microstructure after one and ten cycles, respectively, at an applied stress of 31 MPa (4.5 ksi). In both cases, the resulting microstructures after compaction are very fine. It should be noted that the sample that was cycled ten times shows some coarsening of the carbides. This is a result of the combined effect of strain and time at temperature. By contrast, the structure after 1 hr at 850°C, a temperature that is above the range investigated here, shows marked coarsening as demonstrated in Fig. 10.

This result highlights the importance of using low temperatures and short times in powder compaction where retention of fine structure is desired.

The results presented in Fig. 8 will now be discussed in the light of predictions made in this paper based on transformation superplasticity. In Fig. 2, the useful bounds of stress for enhanced densification of powders through transformation cycling were shown to lie between 1.7 and 30 MPa. The results shown in Fig. 8 support this prediction. The upper bound is evident by noting that the density of the white cast iron compacts is nearly the same, with or without transformation cycling, at stresses close to 30 MPa (Fig. 8). Normal slip processes dominate the densification at and above this upper bound of stress and therefore little benefit is obtained from transformation superplasticity. The lower bound corresponds to a stress of about 1.7 MPa. This stress is considered to be a lower bound because, at and below this stress, even after a large number of transformation cycles, poor total densification is achieved. For example, extrapolation of the data in Fig. 9 to 1.7 MPa, would indicate less than 90% densification is achieved even after ten cycles. This number of cycles requires 80 min of warm pressing. In order to achieve the practical goal of nearly-full densification at 1.7 MPa a number of hours of transformation cycling are required. We therefore consider the stress bounds shown in Fig. 2 to be well justified by the white cast iron powder densification experiments.

The principles of enhanced densification through transformation superplasticity that have been discussed above could find potential applications in other ferrous-base systems. This is because it is usually desirable to avoid high temperatures during the consolidation of rapidly-solidified powders. One example is the case of high-alloyed tool steels where fine carbides and fine

grain sizes are considered beneficial to room temperature properties. These fine structures are inherent in powders that are rapidly quenched. Utilizing the enhanced densification offered by transformation superplasticity, consolidation at temperatures where little carbide or grain coarsening occurs is possible. Another example of a possible application of transformation superplasticity is that of ferrous base materials with a low eutectoid temperature. An extreme case is the Fe-N system. A eutectoid transformation occurs in this system at 590°C and the eutectoid composition is found at 2.35 wt % nitrogen. Thus consolidation of fine structure powders in an Fe-N alloy could be carried out at temperatures lower than in Fe-C alloys. Consequently, negligible coarsening of ultra-fine structures can be expected.

CONCLUSIONS

1. When materials are subjected to an externally applied stress during phase transformations, high strain rate sensitivities and low strength can result. This phenomenon, known as phase transformation superplasticity, can be utilized to enhance densification of powders. The factors affecting the strain that occurs upon transformation are: volume change upon transformation, strength of the phases involved, applied stress and heating-and-cooling rate.

2. Based on this review it is proposed that, for ferrous-based materials, there is a limited range of useful externally applied stresses over which significant total strains (\sim 10%) resulting from transformation can be generated in a small number (< 50) of thermal cycles. This range of useful applied stresses is from 1.7 MPa to 34.5 MPa for ferrous materials that exhibit transformations in the range 727 to 800°C. Below 1.7 MPa, too many cycles for practical purposes are required to generate significant strains, and above

34.5 MPa, normal creep processes dominate deformation.

3. The above prediction has been tested by consolidating white cast iron powders manufactured by rapid solidification processing. It has been shown that significant improvements in the consolidation of powders occurs by thermal cycling through the A_1 transformation temperature (675°C \neq 775°C) during the simultaneous application of a stress in the range 3 MPa to 30 MPa.

4. The benefit of utilizing low temperatures and short times is that fine structures within the powders are retained. This is in contrast to conventional consolidation techniques where the high temperature used will lead to rapid coarsening of the structure.

5. The above conclusion suggests that a method of densification utilizing phase transformation superplasticity is of potential technological significance.

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Fig. 1: Influence of stress on the plastic strain during a two-way phase transformation for a number of metals. The transformation strain is normalized by the volume change upon transformation and the stress is normalized by the hardness of the transforming material [14-18].



Fig. 2: Influence of stress on the transformation strain $(\alpha + \gamma + \alpha)$ for a number of ferrous base materials [11-13]. The dashed vertical lines bracket the stress range where transformation superplasticity (675°C \neq 775°C) can contribute significantly to densification of ferrous powders.



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Fig. 4: Scanning electron micrograph of the warm pressed white cast iron powder after ten two-way phase transformations ($675^{\circ}C \neq 775^{\circ}C$) at a stress of 31.3 MPa. An interdiffusion zone consisting of pearlite bounds the fine-structured white cast iron region and the coarsegrained mild steel plate region.



Fig. 5: Scanning electron micrograph of a powder particle of white cast iron after annealing at 650°C for 15 min. The microstructure consists of a fine cementite structure in a ferrite matrix.

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Fig. 6:

The photomicrographs illustrate the influence of pressure on densification of white cast iron powders after one two-way transformation cycle (675°C \neq 775°C). (A) pressure of 1.6 MPa (<80% dense), (B) pressure of 15.7 MPa (94% dense), (C) pressure of 31.1 MPa (38% dense).



Fig. 7:

Influence of time on the densification of white cast iron powders as a function of stress at 650°C.



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Scanning electron photomicrographs show the fine structure retained in the white cast iron powders after warm pressing under cyclic phase transformation. (A) 1 cycle, 675° C $\approx 775^{\circ}$ C, under 31,3 MPa (4.5 ksi) and (B) 10 cycles, 675° C $\approx 775^{\circ}$ C, under 31.3 MPa (4.5 ksi).

Fig. 9:



Fig. 10: Scanning electron micrograph of white cast iron powders, originally densified at 650°C, then heated to 850°C for 1 h. The cementite particles have coarsened considerably after heating to 850°C.