

INVESTIGATION OF THE SINTERING PROCESS USING NON-CONTACT ELECTROMAGNETIC ACOUSTIC TRANSDUCERS

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ABSTRACT

In-situ characterizations of green state part density and sintering state have long been desired in the powder metal community. Recent advances in non-contact electromagnetic acoustic transducer (EMAT) technology have enabled in-situ monitoring of acoustic amplitude and velocity as sintering proceeds. Samples were made from elemental powders of Al (99.99%), Al (99.7%), Ag, (99.99%), Cu (99.99%) and Fe (99.9%). The powders were pressed in a uniaxial die and examined with acoustic waves for changes in velocity and amplitude during sintering for the samples containing Al, Ag, and Cu. The changes in acoustic properties were correlated with sample microstructures and mechanical properties. Evolution of a series of reverberating echoes during sintering is shown to provide information on the state of sintering, and changes in sintering kinetics as well as having the potential for detection of interior flaws.

INTRODUCTION

The process of sintering is used daily to manufacture complex shaped products with uses ranging from automobiles to home appliances. Although, the practice of sintering is used daily the understanding of the fundamental mechanisms and parameters that govern the process of sintering is limited [1]. Most of the current knowledge base of sintering theory is limited to solid-state sintering of a single component [1]. Moreover, the sintering conditions that are usually modeled are limited to ideal conditions [2]. Assumptions that are made to simplify the model include monosized spherical powder, isotropic surface energy, simple diffusion conditions, and no effect due to the sintering atmosphere. In addition, most sintering models do not take into account the presence of oxides, hydroxides, nitrides, or carbides on the surface of the powder that can influence the mechanisms active during sintering and affect the rate of sintering.

The limitations of current sintering models are a direct result of the limitations of the available techniques used to monitor and characterize sintering. Common techniques that have been used to examine sintering in-situ are dilatometry [1], destructive metallography [1], high temperature mechanical tests [3] and some ultrasonic measurements [4-6]. While these techniques have provided some useful information, they all have limitations. Dilatometry only provides a one dimensional length change as a function of time and temperature. Although some shrinkage is usually observed during the sintering process, it is not required for sintering to take place [1]. Moreover, there are other metallurgical changes that can occur simultaneously with sintering that can complicate the length measurement and thereby confound the results obtained. In contrast to dilatometry, destructive metallography provides direct evidence of the sintering process. However, destructive metallography is very time consuming and expensive. More importantly, destructive metallography cannot be used as a method to monitor or control sintering in-situ or in real time, since cooling of the samples to room temperature and sample preparation is necessary prior to

examination. While high-temperature mechanical tests have provided information not previously attainable with any other technique, the process is still a destructive test.

Although standard ultrasonic measurements show promise as a technique to monitor sintering, they are severely limited by the need for a medium to conduct the acoustic wave into the compact [4, 5]. Although air coupled transducers eliminate the need for a liquid medium to conduct the acoustic wave, previous work has only examined samples at room temperature and not during sintering [6]. The use of non-ultrasonic nondestructive inspection techniques to monitor sintering suffer from their inability to perform inspections below the near-surface region with the exception of X-ray and to some extent pulsed eddy current [7]. X-ray inspection requires extensive shielding of production personnel and expensive film and film processing. While development of pulsed eddy current techniques have lessened the impact of the well-known skin effect, the depth of penetration of the induced eddy currents remains somewhat of an issue [7]. It is generally known that ultrasonic inspection is capable of making density measurements by recording the change in velocity of the sound waves with changes in porosity [8, 9]. However, as previously stated, the use of ultrasonic techniques has been severely limited by the need for a medium to conduct the acoustic wave into the compact rendering this inspection technique unusable for measuring the sintering process without outside influence [4, 5, 10].

It is readily apparent that non-contact nondestructive evaluation of powder metallurgy (P/M) components is a challenge and is not generally practiced in a production environment [11]. However, nondestructive characterization of P/M components is highly desirable; because substantial cost savings and increased reliability can be attained with 100% inspection. In addition, non-contact nondestructive evaluation may provide a greater understanding of the sintering process itself. An increased scientific understanding of the sintering process enables intelligent process control, greater reliability of the production process and a lower rate of rejection of the finished product. In order to realize these potential benefits, it is necessary therefore that a better understanding of the sintering process be based on real time in-situ nondestructive evaluation of P/M samples.

Recent advances in electromagnetic acoustic transducer (EMAT) technology have overcome the need for a medium to conduct the acoustic wave by using the natural electrical conductivity of the metal compact to induce and detect coordinated atom movements. The physical principle underlying the generation of an acoustic wave using an EMAT involves the generation of Lorentz forces. When a wire is placed near the surface of an electrically conducting object and is driven by an alternating current, eddy currents will be induced in a near surface region of the object. If a static magnetic field is also present, these eddy currents will experience Lorentz forces. The forces allow for a wave to be generated without contact between the EMAT and the material to be inspected. Moreover, this technique can be used at elevated temperature and has been shown to provide in-situ real time monitoring of the sintering process [12-14]. The EMAT has other advantages besides high temperature operation and no requirement for couplants. One is the low sensitivity to surface roughness. Since the ultrasonic wave is generated in the sample, surface roughness does not affect the ultrasonic wave to the extent it would for air coupled or standard transducers. Secondly, the use of a transverse or shear wave EMAT allows for the detection of a transient liquid phase, which may be difficult to detect with other techniques. Measurements of the sintering process using the described nondestructive evaluation (NDE) equipment may be used to validate and modify current sintering models.

EXPERIMENTAL PROCEDURE

The powders used in this work consisted of 99.99% pure high pressure gas atomized (HPGA) Al, 99.7% pure HPGA Al, 99.99% pure HPGA Cu, 99.99% pure HPGA Ag, 99.7% pure air atomized Al and 99.9% pure Fe. The HPGA powder was made at the Department of Energy's Ames Laboratory while the air-atomized Al and atomized Fe were purchased from a commercial source. Samples of pure Al, pure Fe, pure Ag and Al-4 wt.% Cu were made by pressing either the pure powders or by blending elemental powders in the correct proportions. The process that the Ames Laboratory used to make the "clean" HPGA powder is described in detail elsewhere [15]. Each type or blend of powder was pressed in a uniaxial die at 414 MPa (30 tsi) to make a disk shaped

sample. In addition, samples of pure Ag and Fe were pressed at a range of lower pressing pressures to obtain a range of initial densities. A die wall lubricant was the only lubricant used in the fabrication of the green samples to eliminate any variations due to binder removal. Except for the pure Fe, all the 4 cm diameter and 6.25 mm thick green samples were placed in a laboratory furnace containing access holes on top and bottom of the furnace for the high temperature EMAT. The furnace and high temperature EMAT were made by Sonic Sensors and are shown in previous work [12-14]. Transverse ultrasonic wave tone bursts with polarization parallel to the flat surface of the P/M compact were created using the EMAT and propagated across the thickness of the compact. These tone bursts consisted of three cycles at 1.25 MHz. Creation of the incident wave and detection of the transmitted echoes was accomplished using a Ritec RAM-10000 pulser receiver in conjunction with the EMAT probes. Individual samples were sintered in a flowing nitrogen or argon atmosphere. While at elevated temperature ultrasonic signals were digitized and stored at various times during the sintering process of each sample type. In contrast, the Fe samples were sintered in a separate furnace and examined at room temperature in the EMAT apparatus. Three types of mechanical tests were performed in this study. A small punch test was used for the 99.99% pure Al samples that exhibited large amounts of ductility. The small punch test apparatus is discussed in detail elsewhere [16-18]. In addition, tensile specimens of the HPGA Al and air atomized Al were made and tested according to MPIF standard 10. The third mechanical test was the transverse rupture strength (TRS) test.

RESULTS

Aluminum

Typical waveforms taken during different stages of sintering of the HPGA Al puck are shown in Figures 1a, 1b, and 1c respectively. The first waveform (Figure 1a), taken during the heating period before the sample reached the sintering temperature (500°C), exhibits very weak echoes. The next waveform, shown in Figure 1b, was taken after 50 minutes at the sintering temperature and shows evidence of 3 distinct echoes. Moreover, the 3 echoes are shifted slightly to shorter time and are higher in amplitude. The last waveform, shown in Figure 1c, was taken after 180 minutes at the sintering temperature. The most notable change in the waveform is the increase in amplitude of the 3 echoes.

A plot of the measured amplitude of the first two echoes at various elapsed times is shown in Figure 2. The zero time represents the time when the sample reached the sintering temperature of 500°C. The amplitude of the two echoes is relatively constant during the time period between 0 and 50 minutes. During the time period between 50 and 100 minutes the amplitudes of both echoes 1 and 2 increase significantly. During the final period between 100 and 210 minutes the amplitude continues to increase, but at a reduced rate. The green density and sintered density of the compact was measured to be 97.3% and 96.3% respectively by simple volume for the green sample and by the Archimedes technique for the sintered sample. The lack of a significant change in density is important, since the observed changes in the ultrasonic waveform are therefore not due to a change in density. In addition, the samples examined are 99.99% pure Al and no second phase or solid solution strengthening effects should exist. The only expected and observed change in the sample during isothermal annealing at 500°C for 210 minutes is the bonding of the powder particles due to sintering with the resulting strength increase being exhibited by the increase in echo amplitude. Examination of the optical microstructures of the sintered and green sample, shown in Figure 3a and 3b respectively, also indicates that significant densification or microstructural changes did not occur.

A similar experiment using air atomized Al powder was also performed at 500°C. In contrast to waveforms taken for the 99.99% pure HPGA Al powder, there was no observed change in the waveform over time at 500°C. The lack of sintering exhibited by the air atomized powder was expected, since air atomized Al powder ordinarily has a relatively thick aluminum oxide coating that is formed during processing to reduce the explosive nature of aluminum powder.

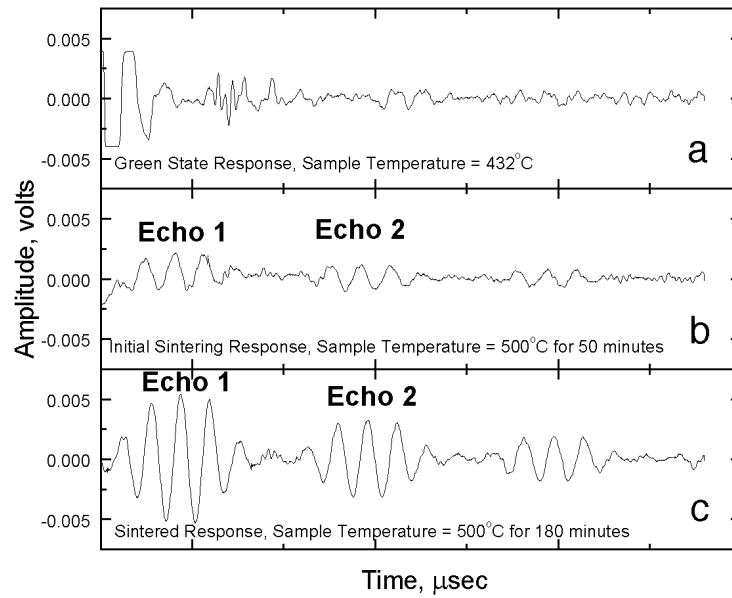


Figure 1. Typical ultrasonic waveforms taken during sintering of the 99.99% pure HPGA Al sample at 500°C.

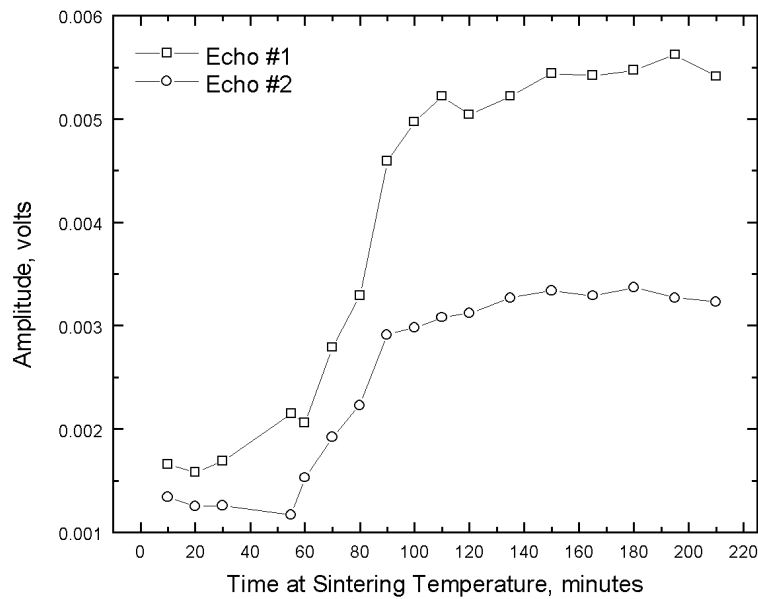


Figure 2. Measured amplitude of echo 1 and echo 2 at the various elapsed times during sintering of the 99.99% pure HPGA Al sample.

Four new 99.99% pure HPGA Al samples were made and sintered for various times. The amplitudes of the first two echoes in the acoustical signal along with the measurement of the acoustic velocity for each of the new samples are shown in Table 1. The data shown were taken while the sample was at room temperature after the sample had cooled. From Table 1 it is apparent that the measured velocity for each of the samples is nearly the same, while the peak amplitude varies. The nearly identical velocity values indicate that the nominal densities of these samples are the same, although there are likely variation in density throughout the parts.

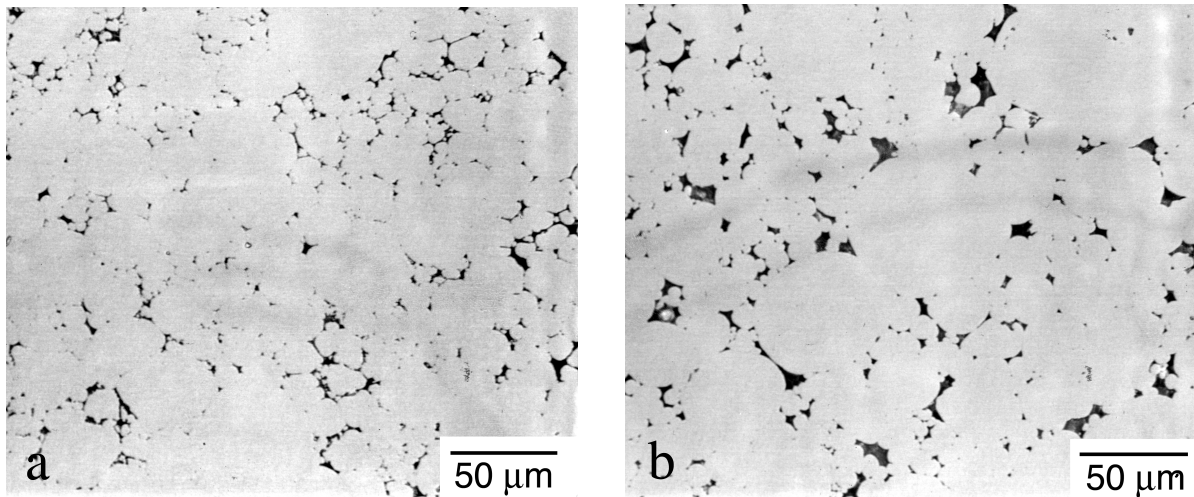


Figure 3. Optical micrographs of 99.99% pure HPGA Al sintered at 500°C for 180 minutes (a) and 99.99% pure HPGA Al green compact (b).

Table 1

Sample #	Peak#1 Amplitude (V)	Peak#2 Amplitude (V)	Velocity (M/s)
1	0.0240	0.0219	3047
2	0.0205	0.0181	3005
3	0.0184	0.0161	2997
4	0.0141	0.0113	3041

Three small punch test disks were made from the four different samples and tested to determine a relative strength of the different sintered compacts. A plot of the maximum load versus echo amplitude of the first echo is shown in Figure 4 for the four different samples of 99.99% pure HPGA Al sintered for various times. From this figure it is apparent that there is a linear relationship between the maximum load and the echo amplitude measured. The linear fit of the data yields a correlation coefficient (r^2) value of 95%, which is very reasonable.

In-situ sintering experiments were conducted using the 99.7% pure HPGA Al powder to investigate the change in sintering kinetics related to changes in the sintering temperature. The peak-to-peak amplitude change with time of echo #1 is shown in Figure 5 for sintering temperatures of 500°C, 525°C and 550°C. As expected the rate of sintering increased with increasing temperature. However, a dramatic increase in the apparent sintering response of the 99.7% pure HPGA Al sample was observed at 550°C. The immediate onset and enhanced amplitude response, with only a 25°C increase in temperature from 525°C test, suggests a significant change in the diffusion mechanism(s) controlling the sintering rate.

To investigate whether the change in sintering mechanism at 550°C is also active in air atomized powder a compact made from 99.7% pure air atomized powder was measured. The typical ultrasonic waveforms taken during sintering of the 99.7% pure air atomized Al sample at 550°C are shown in figure 6. The appearance of ultrasonic echoes in the 99.7% pure air atomized Al sample indicates that air atomized powder can be sintered above 550°C. However, there are apparent differences in the maximum ultrasonic amplitude obtained when compared to the 99.99% pure HPGA Al.

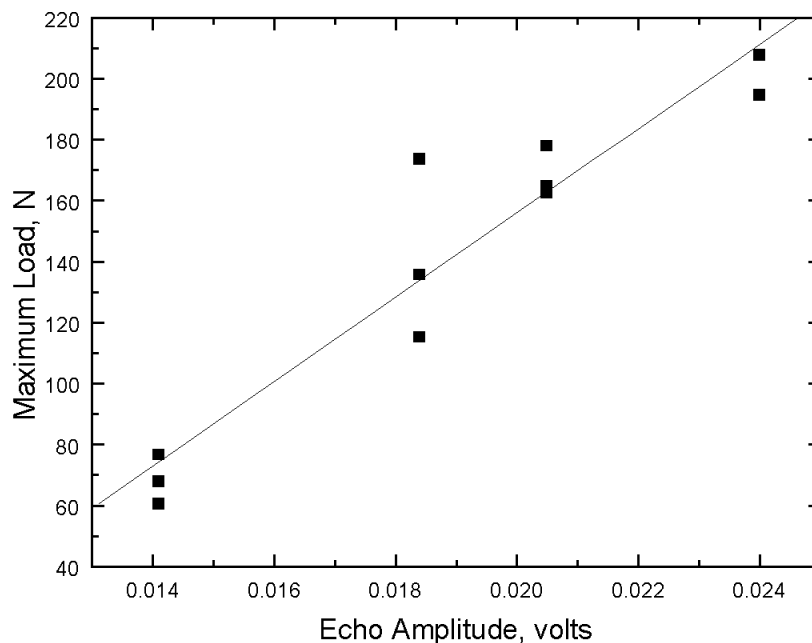


Figure 4. A plot of the measured maximum load obtained in a small punch test versus echo amplitude of echo 1 for four 99.99% pure HPGA Al samples.

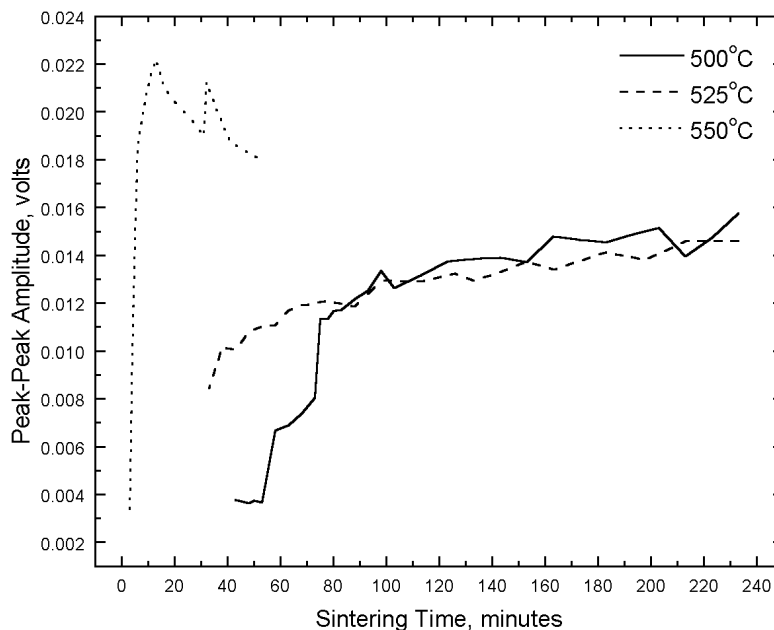


Figure 5. Sintering response of 99.7% pure HPGA Al with changes in temperature.

Two tensile samples made from 99.99% pure HPGA Al and 99.7% pure air atomized Al were pressed and sintered at 550°C for 1 hour. The resulting stress strain curves for the four samples are shown in Figure 7. It is readily apparent that the 99.99% pure HPGA samples are more ductile and exhibited a lower yield and tensile strength than the 99.7% pure commercial air atomized (CAA) Al. Moreover, it should be mentioned that the CAA samples both fractured in the grip

section, while the HPGA samples broke in the gage section. The second HPGA sample exhibited a significantly lower elongation and ultimate tensile strength. Examination of the second HPGA sample indicated that it failed due to a defect in the pressed sample.

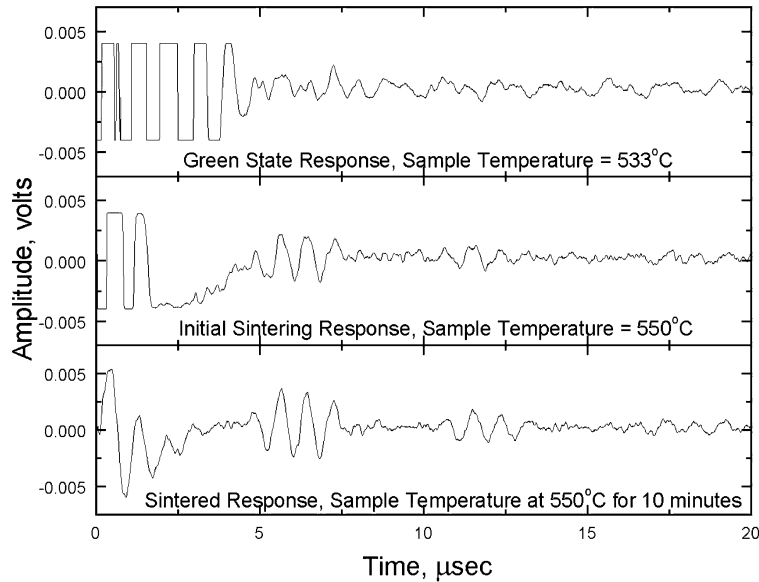


Figure 6. Typical ultrasonic waveforms taken during sintering of the 99.7% pure air atomized Al sample at 550°C.

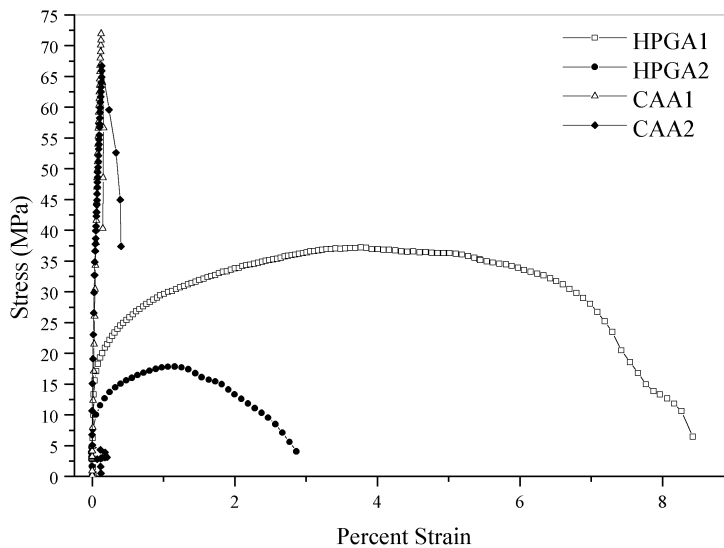


Figure 7. A plot of the measured stress versus strain for two tensile samples made from Ames Laboratory high pressure gas atomized (HPGA) Al and commercial air atomized (CAA) Al sintered at 550°C for 1 hour.

Figure 8 is a plot of the measured amplitude of echo 1 and echo 2 at the various elapsed times during sintering of HPGA Al-4wt% Cu elemental blend sample. The effect of the transient liquid phase around 20 minutes on the echo amplitude is clearly seen. In addition, a large increase in echo amplitude associated with lowering the sample temperature only two degrees after 63 minutes is also easily seen. This is an indication that some fraction of equilibrium liquid was still present at the sintering temperature.

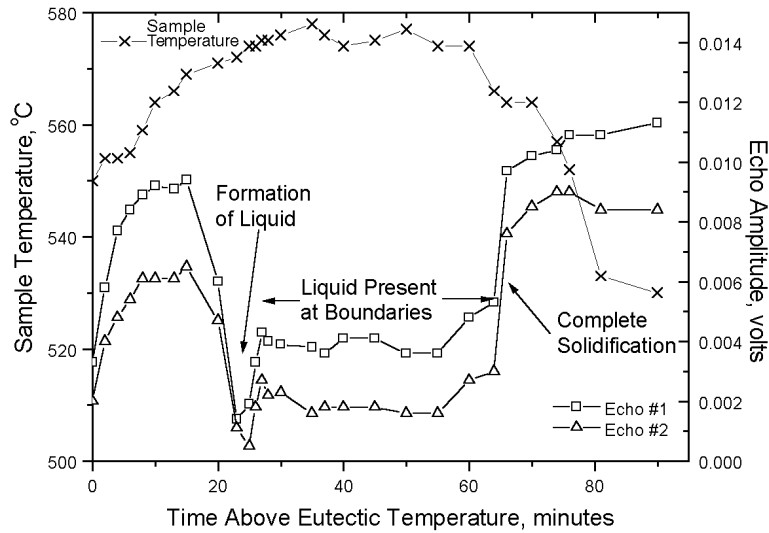


Figure 8. A plot of the measured amplitude of echo 1 and echo 2 at the various elapsed times during sintering of HPGA Al-4wt%Cu elemental blend sample.

Copper

A plot of the measured amplitude of echo 1 and echo 2 at the various elapsed times during sintering of a pure Cu sample at 600°C are shown in Figure 9. In contrast to the pure Al samples, a distinct echo is observable in the green pure Cu sample. Moreover, the behavior of the echo #1 and echo #2 are quite different than what is observed for pure Al. Instead of increasing with sintering time, the echo amplitude of echo #1 increases then decreases before increasing to a maximum value all before reaching the sintering temperature. Then the amplitude decreases to a midlevel value.

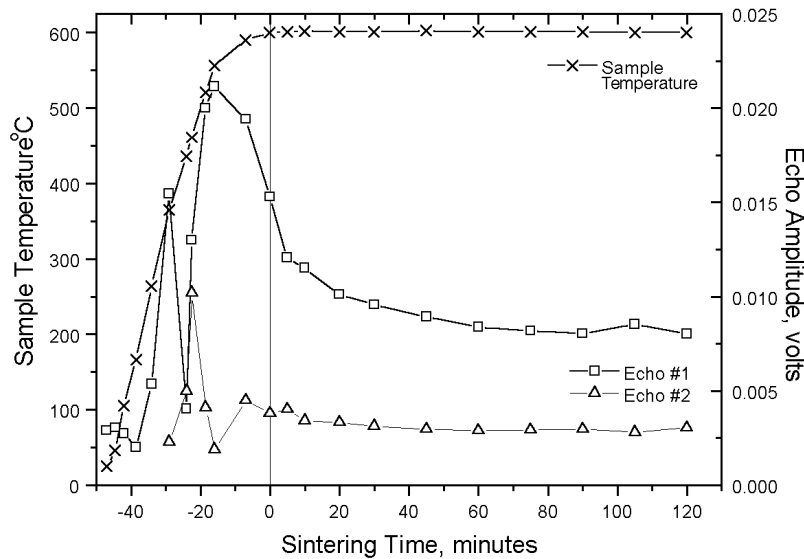


Figure 9. A plot of the measured amplitude of echo 1 and echo 2 at the various elapsed times during the sintering of a pure Cu sample.

A plot of the measured transit time at the various elapsed times during sintering of pure Cu at 600°C is shown in Figure 10. From Figure 10 it appears that the transit time decreases rapidly before reaching the sintering temperature of 600°C. After reaching the sintering temperature the

transit time decreases, but at a much lower rate. It should also be noted that there is an abrupt increase in transit time before the sample reached the sintering temperature of 600°C.

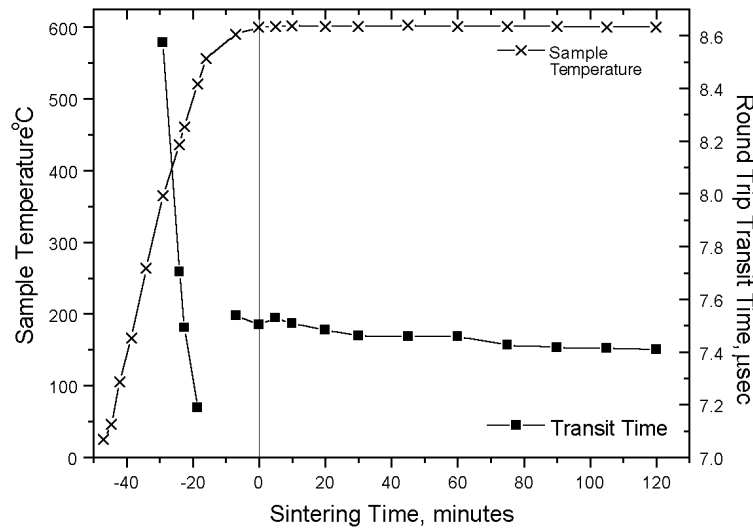


Figure 10. A plot of the transit time behavior of pure Cu sintered by solid state sintering.

Silver

A plot of the measured amplitude of echo 1 and echo 2 at the various elapsed times during sintering of pure Ag at 600°C are shown in Figure 11. Similar to the pure Cu sample, distinct echoes are observable in the green pure Ag sample. However, more echoes are observed in the pure Ag sample. It is interesting to note that the echo amplitude seems to be affected by the sample temperature and not the sintering time.

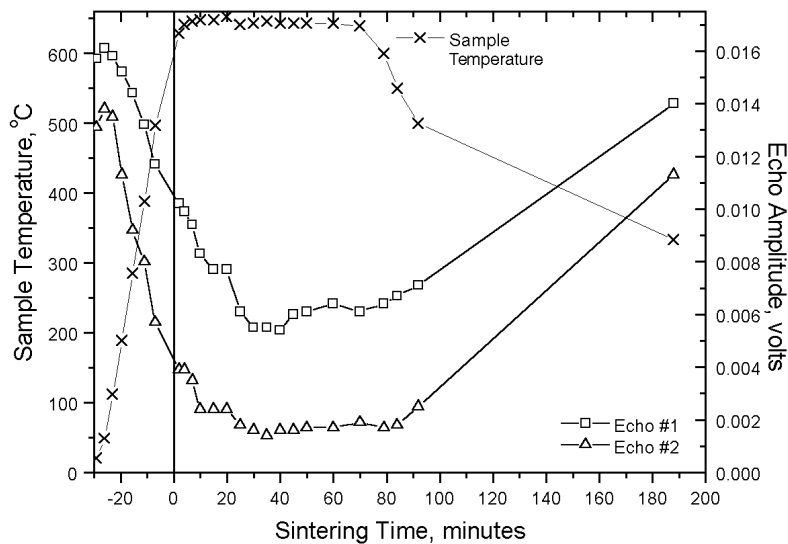


Figure 11. A plot showing the measured amplitude of echo 1 and echo 2 at selected times during the sintering of the pure Ag sample.

A plot of the measured transit time at the various elapsed times during sintering of pure Ag at 600°C is shown in Figure 12. Except for an unexplained decrease and subsequent increase in transit time, the decrease in transit time with time is what would be expected of a sample that is increasing in density with time.

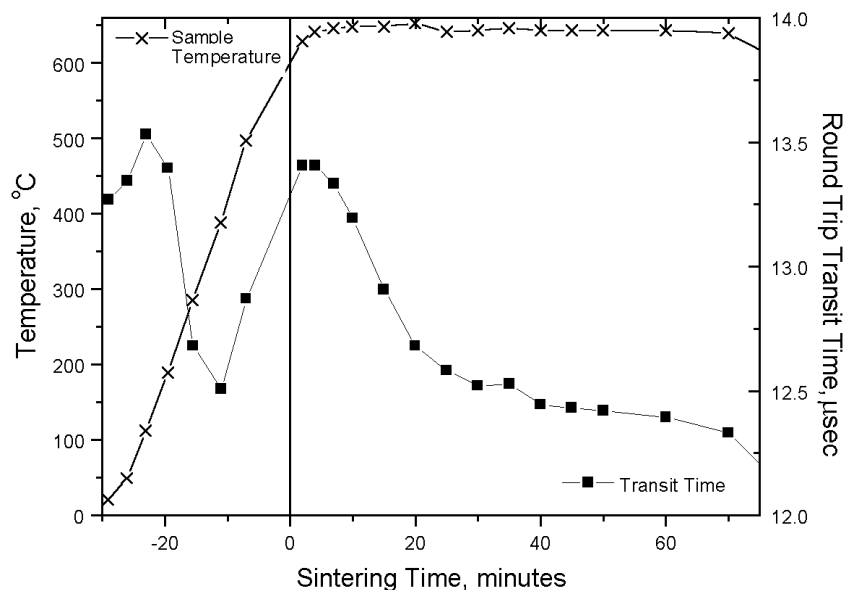


Figure 12. A plot of the transit time behavior of pure Ag sintered by solid state sintering.

Results of the velocity measurements on both the green compact and sintered silver samples at room temperature are shown in Figure 13. Note, the data obtained for the silver sample, pressed at the lowest pressing pressure, exhibited a low signal to noise ratio, preventing accurate determination of the shear velocity. As expected there is a linear relationship between shear velocity and theoretical density of the sintered samples. The measured shear velocities of the green samples are much lower than the sintering samples.

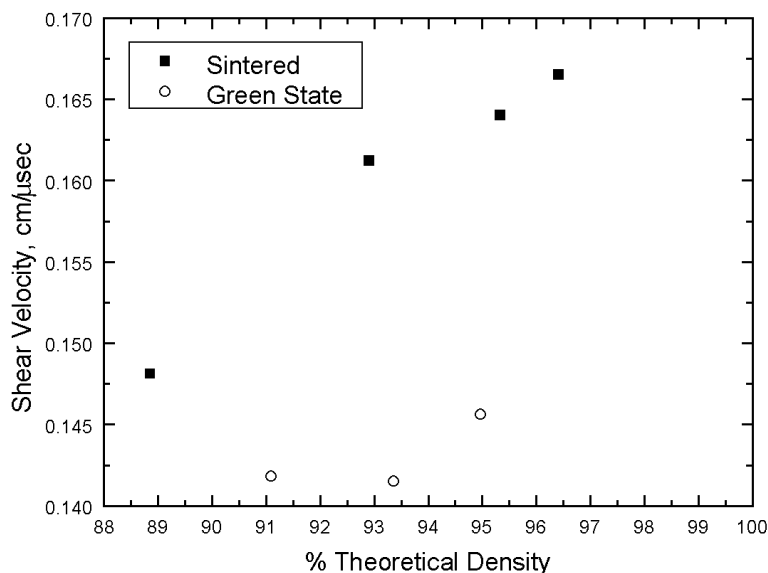


Figure 13. A plot of the variation of ultrasonic shear velocity with measured density of the Ag samples.

Iron

Measurement of the ultrasonic properties of the iron powder compacts was made before and after sintering. The property of primary interest was the ultrasonic velocity since it has been shown to be a relatively clear indicator of the amount of porosity in a material [8, 9]. Results of the velocity measurements on both the green compact and sintered sample are shown in Figure 14 along with the accepted value for the shear velocity in pure iron [19]. An excellent linear correlation is observed between the density and shear velocity measurements for the sintered pucks and the reported value for bulk material.

Longitudinal velocity values were also obtained using contact transducer methods for comparison with standard accepted values. A comparison between the values obtained in this work and those published elsewhere [8] indicated very similar behavior. Although the values obtained in this work are higher than those published elsewhere, the sensitivity of the velocity to changes in the density is very nearly identical in the two cases.

The ultrasonic velocity has also been used previously in a correlation with the strength of as-sintered specimens for 0.65% carbon steel [8]. A similar correlation, shown in Figure 15, was obtained in this work between the shear velocity as obtained using EMATs on sintered iron samples and the maximum force values obtained from a standard transverse rupture strength (TRS) test. The correlation was done using the maximum force rather than the traditional transverse rupture strength due to the rather large ductility observed in the samples possibly causing invalid transverse rupture strength values. A reasonable linearity is observed between the two measurements.

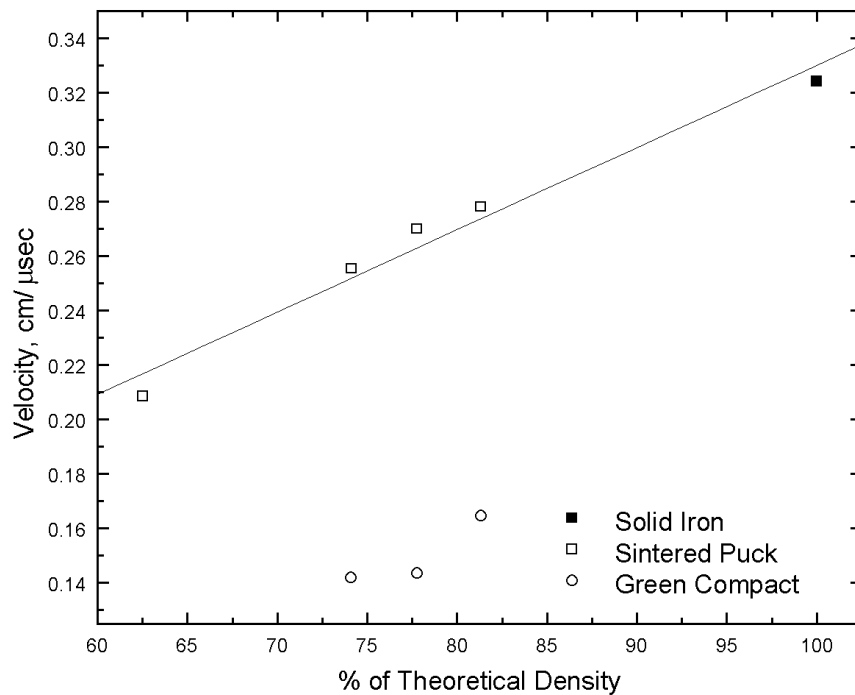


Figure 14. A plot of the variation of ultrasonic shear velocity with the measured density of the Fe samples.

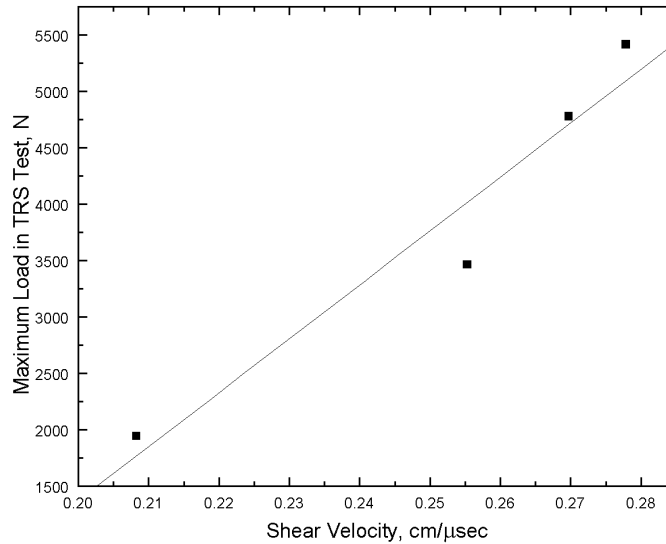


Figure 15. A plot showing the correlation between ultrasonic velocity and TRS maximum load.

DISCUSSION

The acoustic amplitude change shown in Figures 1 and 2 shows a behavior pattern consistent with the expected behavior of HPGA Al during solid state sintering [15]. Initially, the acoustic waves are barely visible in transmission through the sample. At this stage, the sample consists almost entirely of mostly spherical powder particles merely pressed together with possibly a small amount of cold welding. Transmission of acoustic energy through a P/M compact depends on the degree of contact between individual particles. Acoustic transmission is poor in this initial condition, because the contact is mostly weak mechanical bonds. As sintering begins, the acoustic energy also shows a change to a more conventional echo train pattern consistent with transmission of the energy through a medium with stronger bonding. The increase in the echo amplitudes with time further verifies the progress of the diffusion across the powder particle boundaries, thereby strengthening the contact between particles and creating a larger path that improves transmission of the acoustic energy. As the sintering rate begins to taper off, the echo amplitudes rise at a much lower rate trending to a constant slope.

The potential of the described ultrasonic technique is made very evident when the two nearly identical microstructures of the sintered and green microstructures shown in Figure 3 are examined. From the micrographs, it is nearly impossible to distinguish between the two samples since there has been no apparent density change and the pore structures are nearly identical. However, the ultrasonic signals obtained from the two different samples are vastly different and unique. In addition to the sensitivity of the technique, the apparent relationship between echo amplitude and mechanical strength, shown in Figure 4, also demonstrates another useful attribute of the technique. Although additional work is needed to examine the effects of initial density, powder size distribution and temperature on the apparent echo amplitude to strength relationship, the results are extremely encouraging that a nondestructive technique may be used to rapidly determine relative mechanical strength of Al P/M components. Moreover, such a nondestructive technique may be used as a process control during industrial sintering.

Figure 5 shows three such response curves taken at different temperatures for the 99.7% pure Al. From the three response curves shown, it is obvious there is a large difference in the response of the 99.7% purity Al sample sintered at 550°C. The dramatic change in sintering response with only a 25°C change in temperature is an indication of a change in the mechanism controlling the sintering rate. The 550°C sintering temperature is higher than most of the eutectic temperatures found in the aluminum binary phase diagrams suggesting the possibility of liquid formation since impurities exist in the material. If present, any liquid would of course create significant alteration in the sintering response. However, liquid formation is unlikely for these powders since the

compact was made from atomized rapidly cooled powder with only 0.03% impurities. Moreover, 0.03% is well within the solid solubility limits of most if not all of the likely impurities in aluminum.

The significant shift in the response curve to much shorter times as the temperature is increased from 525°C to 550°C appears to correlate with information developed earlier [15] on the decomposition reactions that occur on the surface of the aluminum powder. Anderson et al. [15] showed that as the temperature increased above about 540°C, the final decomposition of aluminum oxide monohydrate surface layer to anhydrous alumina occurred. It is possible that reduction of the thin oxide layer typically found on aluminum powders made by the HPGA process to the anhydrous state may affect the coherence and degree of continuity in the oxide. If either the coherence or degree of continuity in the oxide were degraded sufficiently, the initial break through of the oxide layer would enable faster sintering kinetics.

Earlier work [15], has shown that, in contrast to HPGA Al, commercial quality air atomized Al will not sinter at 500°C. The lack of sintering at 500°C for air atomized powder is attributed to the thick oxide formed during air atomization. The inability to sinter at 500°C was verified using in-situ nondestructive measurements of the sintering response [13, 14]. However, as the results in Figure 6 clearly illustrate, an increase in the sintering temperature to 550°C alters the sintering behavior drastically through changes in the hydration level of the oxide layer. These results indicate that the decomposition temperature of the monohydrate is a very important consideration in the powder consolidation processing of any Al-based alloy. Moreover, these results demonstrate the need for examination of alloy effects on the monohydrate decomposition temperature. It should be also stated, that while the air atomized powder can be solid state sintered at 550°C, the properties are not identical to the HPGA Al. Clearly, the oxide surface greatly affects the final mechanical properties along with the sintering characteristics.

In addition to the previously discussed benefits of the sintering response plot, shown in Figure 5, the data is also conducive to the calculation of an “effective” activation energy. Such a calculation was done using the times necessary for the acoustic peak-to-peak amplitude to achieve the 0.010 volt level. The calculation was done using the 500°C and 525°C temperatures only since the 550°C response appears to be dominated by a different mechanism. Using only the two temperatures clearly limits the accuracy of the calculation, but it is understood that this is only a preliminary attempt at obtaining the activation energy. Using an Arrhenius plot of $\ln(\text{time})$ vs. $1/\text{Temperature}$ with a straight line drawn between the points plotted at the chosen times, the slope of the line was used to calculate the activation energy. The “effective” activation energy obtained from this calculation is 117 kJ/mole for the two temperatures considered. The value obtained appears to be reasonable since the corresponding value for volume diffusion of aluminum is 145 kJ/mole.

The ultrasonic responses of the pure copper, silver and iron samples are noticeably different than what has been shown to exist in pure aluminum. First, the ability to observe ultrasonic echoes in the green compacts made with copper, silver and iron indicates that there are not as many scattering sources in the compacts as there are in aluminum compacts. It is possible that the difference in scattering may be due to the difference in the nature of the particle surface that forms on aluminum versus copper, silver and iron. For example, aluminum has a very tenacious oxide that is hard to break through during the pressing operation, which would limit the number of cold welds made between the particles and therefore reduce the pathways for sound to propagate. In contrast, silver is relatively noble and appears to form more cold welds that enable the propagation and observation of multiple echoes. The apparent effect of powder surface on sintering characteristics should be taken into account in any future models, since the powder surface is not considered in any of the current sintering models [1]. Second, the ultrasonic amplitude behavior of the Cu and Ag samples, shown in Figures 9 and 11 respectively, is not the same as the characteristic and repeatable behavior of the pure Al samples shown in Figure 2. Moreover, the ultrasonic amplitude behavior of the Cu and Ag samples is significantly different from each other. The Cu sample ultrasonic amplitude increases with temperature and then decays. In contrast, the ultrasonic amplitude of the Ag sample decreases as the temperature is increased and then increases again when the sample is cooled. The unpredictable behavior of the ultrasonic

amplitude in the Cu and Ag samples indicates that a mechanical property relationship similar to that illustrated in Figure 4 for Al likely does not exist for Cu and Ag.

Another apparent difference in sintering behavior is exhibited in plots of the transit time as a function of sintering time for the Cu and Ag samples. While the unusual behavior of the transit time before the Cu and Ag samples reach the sintering temperature of 600°C is not understood, the decrease in transit time with sintering time indicates a difference in sintering behavior. The Cu sample only exhibits a small change in transit time during the 120 minutes of sintering at 600°C indicating that the sample isn't significantly decreasing in thickness or increasing in stiffness with time. In contrast, the Ag sample exhibits a significant change in transit time that tapers off with sintering time. The transit time is affected by the thickness of the sample and the sound velocity. Assuming the thickness of the sample isn't changing dramatically, the primary reason for the change in transit time is the increase in sound velocity due to the increase in stiffness of the sample. This increase in stiffness is due to the mechanical bonds of pressing becoming metallurgical bonds and some change in density. However, Figures 13 and 14 demonstrate that the change in velocity due to densification is a much smaller effect than the difference in the type of bonding.

Similar to the Ag results shown in figure 13, the results of the iron studies were conclusive in the ability to discriminate between samples with density variations following sintering as shown in Figure 14 and confirming the relationship reported earlier by Papadakis [8]. However, extension of the ability to estimate the density from the acoustic velocity into a measurement of the strength as determined through the measurement of the acoustic velocity has been confirmed in this work. While the temperature limitations of the current sintering apparatus preclude actual in-situ Fe measurements, the similarity between the results shown in Figure 13 and 14 indicate that Ag may be used as a model system for Fe.

In addition to providing information about the solid state sintering the technique shows promise in detecting small fractions of liquid formed during sintering operations. The EMAT system being used is capable of generating either longitudinal or shear waves with shear waves being the mechanism of choice for characterizing liquid phase or transient liquid phase sintering. Shear waves are generally not transmitted well through liquid media allowing the detection of a liquid phase at the grain boundaries that is present during sintering of most commercial powder metallurgy Al alloys. The NDE technique described in this paper has been shown to be very sensitive to relatively small (<2%) percentages of liquid formation, which may be unobserved in other characterization techniques such as high temperature X-ray diffraction. Accurate assessment of the total time a transient liquid phase is present in a real sample is beneficial in the understanding of liquid phase sintering.

CONCLUSIONS

The real-time in-situ non-contact monitoring of the sintering process has been demonstrated with an ultrasonic nondestructive evaluation method using high-temperature Electromagnetic Acoustic Transducers. The change in ultrasonic waveforms observed is a direct manifestation of the bonding that occurs during sintering. Solid state sintering of Al is possible even when a thick oxide layer is present as long as the sintering temperature is higher than the decomposition temperature of the monohydrate. The possibility of using this system for determination of the "effective" activation energy of sintering for various systems has been demonstrated. Further work will be necessary to determine the relevant contributors to this activation energy since it appears that much more will be involved than simply volume diffusion of the materials. The kinetics of transient liquid phase sintering is now observable with the new method enabling determination of critical processing parameters. Use of the EMAT system for prediction of the strength of sintered parts has also been shown to be in reasonable agreement with earlier published works. Moreover, additional direct measurements of the sintering process using the described NDE equipment may be used to validate and modify current sintering models. With further development of the EMAT to withstand higher temperatures, an adjustable sintering process control system of Fe-based P/M components could be manufactured.

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