Reprinted from:
Metal and Ceramic Injection Molding
Advances in Particulate Materials - 1994
Volume 4
Metal Powder Industries Federation, Princeton, N.J.

IMAGE ACQUISITION AND ANALYSIS DURING SINTERING: A METHOD OF MONITORING AND CONTROLLING P/M PART CHARACTERISTICS

David W. Yoel, Charles W. Miller, Jr., Robert K. Olson

Centorr/Vacuum Industries, Inc.
Nashua, NH 03063

ABSTRACT

Dimensional measurements of powder metal and metal injection molded part dimensions are made by part imaging during debinding, presintering and sintering to optimize process control. A digital image acquisition and control system is now under development that acquires a sequence of microscopic and macroscopic images during processing, enhances the images, compares the dimensions of the part as the cycle progresses, and correlates changes in part dimensions to process parameters including temperature, pressure, gas composition, etc. Initial experiments demonstrating the imaging techniques are presented. Its use in statistical process control and as a real-time method for controlling and improving quality are discussed.

INTRODUCTION

Producers of Powder Injection Molded (PIM) parts are constantly striving for improved quality and lower manufacturing costs. Inspection before and after each step in the cycle is routine. Inspection techniques include physical measurements of part dimensions using mechanical instruments (e.g., calipers), and visual inspection to detect flaws such as flash, inconsistent surfaces, or incomplete filling in green parts. Recent work in the field has focused on automating this visual inspection using digital imaging techniques (Ref. 1). In high volume production, these measurements can be analyzed using Statistical Process Control (SPC) methods to control quality, consistency, and yield, and to lower the cost per part produced.

New instrumentation is also under development to make measurements during powder production, premixing, mixing, and in the injection molding phases of the manufacturing cycle. These are *in-situ* methods that include eddy current, pressure, and ultrasonic sensing techniques (Ref. 1, 2, 3). Each of these sensing methods examines a different process parameter. Eventually, the successful techniques will meld into a complementary set that will contribute measurements to an "intelligent control system".

The concept of Intelligent Processing of Materials (IPM) involves combining *in-situ* sensor data with process models and process knowledge in an intelligent controller to facilitate on-line design and control of process cycles to achieve direct control of part characteristics (Ref. 4). Fundamental to IPM is the measurement of actual part characteristics rather than process parameters such as temperature and pressure. IPM controllers "feed-forward" and "feed-back" continuously updated process cycles to

traditional sensor-controller sets (e.g., thermocouple-programmable PID controller pairs) to optimize part characteristics. A critical aspect of any intelligent furnace control system is the development of effective and non-invasive sensing methods.

Quality Control (QC) methods depending exclusively on final inspection of sintered parts, result in lower yields, and a higher cost per part than techniques that measure part characteristics further "upstream" in the manufacturing cycle. Motivation therefore exists for inspecting parts before the completion of the sintering step. Researchers have been developing *in-situ* measuring techniques for high temperature processes for many years. Thermogravimetric Analysis (TGA), for example, has been effectively used in PIM debinding studies (Ref. 5). IPM systems have been under development for other types of high temperature processing as well, but little or no effort has been applied to date in PIM sintering.

This paper discusses a new method of making direct measurements of the physical dimensions of PIM parts during the sintering cycle. First, the approach and experimental setup are described. Images acquired during sintering and their analysis are then presented. The paper concludes with a discussion of the applicability of the technique to present and future (intelligent) PIM manufacturing systems.

APPROACH

Debind/Presinter/Sinter Method Studied:

The method of debinding, presintering, and sintering selected for evaluation was the combined "Injectavac™" batch process cycle developed by Centorr/Vacuum Industries (Ref. 6). This method removes binders, presinters and sinters PIM parts in a single process cycle. Paraffin wax binders are first sublimated at low temperatures and carried to a vacuum pumping system designed to pump gas streams containing particulates and resins. At slightly higher temperatures, polymer breakdown products are pumped away. As the temperature increases through presintering and sintering, a partial pressure of inert gas is maintained over the parts to eliminate the sublimation of high vapor pressure constituents (e.g., chromium). An entire Injectavac® process cycle typically requires 15-20 hours to process loads to 500 Kg (1100 lb.). These systems generally use graphite hot zones (Ref. 5) and are used to process nickel-irons, steels, and stainless steels (including 316L and 17-4PH).

In-Situ Sensing Technique Selection:

The first issue addressed in this work was to determine what measurements could be made without perturbing the thermal environment of the part, and what value such measurements would have in improving process control and part quality. A critical requirement was that the parameter sensed be an actual part characteristic rather then a process parameter. A second requirement was the ability to use the technique during the entire thermal processing cycle including debinding, presintering, and sintering, The third requirement was that the technique have the potential for use in a production system so as to enable the possibilities of IPM in the future.

Methods considered included dilatometry (to measure the physical dimension along one axis), thermogravimetric analysis (to measure mass), and image analysis (to measure dimensions along at least 2 axis). Table 1 summarizes this comparison. Dilatometry was not utilized because of concerns that intricately shaped parts would be distorted by the sensing mechanism and because it would not be effective during debinding since no shrinkage occurs during this stage in the cycle. Although TGA is nonperturbing, and it is useful during debinding, (Ref. 5), its use during sintering is limited.

The final method considered was the visual measurement of part characteristics. As mentioned earlier, physical measurements of part dimensions at room temperature are a normal part of PIM process inspection. Implicit in making these physical measurements is direct visual observation. Visual observation has been a highly effective tool for accelerating process development in other areas of materials processing including crystal growth, brazing, and welding (Ref. 7). Visual observation of the melting of fluxes

and filler metals, correlated to temperature measurements, has recently been used in quality control in brazing as well. Because it has a demonstrated value in defining part quality, it is noninvasive, it is useful throughout the process cycle, may be used to observe blistering, warpage and incomplete filling, and it may be used to optimize cycle time, visual measurement was the method selected.

Several problems arise when attempting optical measurements in a high temperature vacuum environment. These include; 1) obtaining visual access; 2) minimizing pertebations to the thermal environment; 3) maximizing the magnification (given the large working distances typically required to maintain the imaging system below its maximum operating temperature),; 4) acquiring quality images in a high temperature environment where the infrared radiation emitted by the system tends to saturate imaging equipment, and; 5) making quantitative measurements from the visual information. These problems can only be resolved by modifying the design of both the thermal processing equipment and the imaging system.

Thermal Processing System Selection:

One method of acquiring visual access, minimizing perturbations, minimizing the working distance, and limiting infrared emissions is to utilize a "transparent furnace" (Ref. 7). Although a program is currently underway to increase their temperature rating, transparent furnaces are currently limited to temperatures below 1000°C, and so are not useful for MIM processing at present. A simpler approach was to adapt a more traditional vacuum furnace for enhanced optical access.

EXPERIMENTAL SETUP

Image Acquisition and Analysis:

A variety of visual monitoring methods were considered including direct human observation, still photography, and standard video. Digital image analysis is the preferred technique because it is fast, quantitative and accurate within the limitations of standard analog video. Digital image acquisition and analysis systems are also inexpensive.

The system developed is described schematically in Figure 1. The actual hardware implementation is shown in Figure 2. A "Cool Window" (by Centorr/Vacuum Industries), located in the furnace viewport, reflects infrared energy back into the hot zone while allowing visible light to transmit through. This assures that the thermal environment in the hot zone is not perturbed, a critical requirement. This Cool Window also has the advantage of minimizing the heat flow out of the furnace. Since lenses are not generally designed for high temperatures, the radiant thermal energy from the furnace would otherwise be sufficiently intense that it could damage the lens. The lens was selected to magnify the image of the part at a large working distance. The magnified optical image is acquired by a silicon charge-coupled-device (CCD) camera and transmitted to a computer. The computer stores the image data in memory and displays it on the monitor. The data is stored as a two-dimensional array of "grey" (or brightness) levels. Once in memory, the image can be enhanced. For example, dark images can be brightened, faint edges can be sharpened, etc. The distance between any two points in the array can be determined as a function of the x-y coordinates of each point. Sequences of images can be displayed, and one image can be subtracted from another to display the difference between them. In the case of MIM sintering, this method can be used to quantify the shrinkage of the part being processed.

Furnace:

A commercially available front opening, high temperature controlled atmosphere/vacuum furnace was utilized, a Testorr™ furnace manufactured by Centorr/Vacuum Industries, Nashua, NH. A quartz viewport with a 2" wide by .75" tall view path was positioned on the centerline of the furnace hot zone. This viewport provided an 8.5" working distance between the specimen and the optics. Filters were placed between the optics and the viewport to reduce

infrared transmission to the CCD camera. In order to visually monitor the component from room temperature to sintering temperature an illumination system was required. This illumination source was integrated with the vacuum chamber on the vertical centerline and at 90°C to the optical sight axis.

Material Processed:

The material processed was a 17-4PH stainless steel part with 40% paraffin wax/polymer binder. These parts have been successfully processed by Centorr/Vacuum Industries using the Injectavac™ process cycle.

Experimental Sequence:

The physical dimensions of the green part was first measured. Once the green dimensions were known, the part was installed in the furnace. The initial image acquired before the thermal cycle calibrated the array. Since the working distance was not changed and the lens magnification was not adjusted, this calibration was valid throughout the thermal cycle. As the part shrank, the distance between two defined points on its edge changed, and the coordinates of these two points in memory changed. Since it was the difference that defined the dimension of interest, this shrinkage could be quantified.

EXPERIMENTAL RESULTS AND ANALYSIS:

Figures 3 and 4 show the green part installed in the furnace as seen on the computer monitor. The part was illuminated by a white light source. Four dimensions were measured before installation, as shown in Figure 5. The original dimensions were A = .060", B = .280", C = .142" and D = .232". These measurements were used to define the computers reference dimensions. Figure 6 shows the part at 1000°C at the end of a presinter soak. The sharply defined edge of circle D was used to fit a circular object on the image (Figure 7). The diameter of that circle, D = .232", demonstrates that the part had not changed in size. As the temperature increased to the sintering temperature, the CCD camera was overwhelmed with infrared energy and additional filters were required. Although image quality suffered, quantitative measurements were still possible. Figure 8 shows an enhanced image acquired after approximately 45 minutes at the 1200°C sintering temperature. This is an enhanced image, compiled from two separate images acquired in sequence. At this point, the part had begun to contract, with A = .057" and C = .136". This indicated that the linear dimensions had decreased by approximately 5%. Twenty-five minutes later (Figure 9), the diameter D = .210". The linear dimensions were now approximately 10% smaller. Figure 10 shows the part again after the furnace has cooled to room temperature. Dimensions are A = .055", B = .243", C = .124" and D = .205". This indicates the linear dimensions were approximately 13% smaller. The part was removed from the furnace and the measurements confirmed using calipers.

SUMMARY AND CONCLUSIONS

These preliminary experiments demonstrate that visual monitoring can be used to observe the sintering of MIM parts. It is shown that part size versus time during sintering can be quantitatively measured. Part sagging can also be seen. Image filtering and part illumination must be improved to optimize image quality, leading to improved resolution measurements.

ACKNOWLEDGEMENTS

Special thanks to Sheldon Kennedy of Centorr/Vacuum Industries for advice on sintering and Steven Bates of Thoughtventions Unlimited for his suggestions on imaging.

TABLE 1

CRITERIA	DILATOMETRY	TGA	VISUAL
Perturbs Parts?	Yes	No	No
Perturbs Thermal Environment	Yes	No	No
Effective During Debind	No	Yes	Yes
Effective During Sinter	Yes	No	Yes
Potential For Use In Production	No	No	Yes

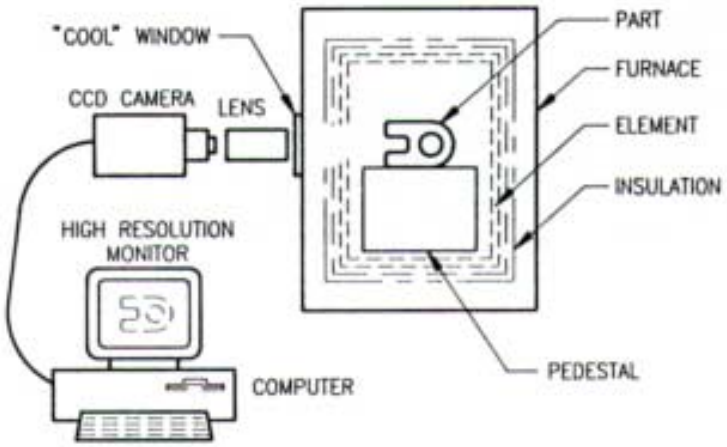

Figure 1. Sketch of experimental setup.

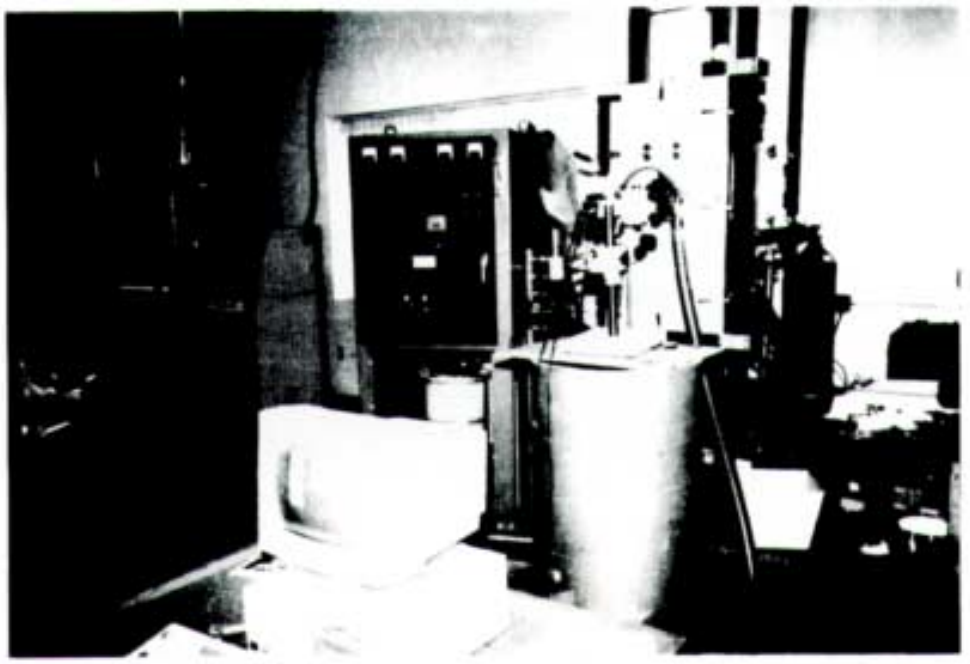

Figure 2. Laboratory implementation of system.

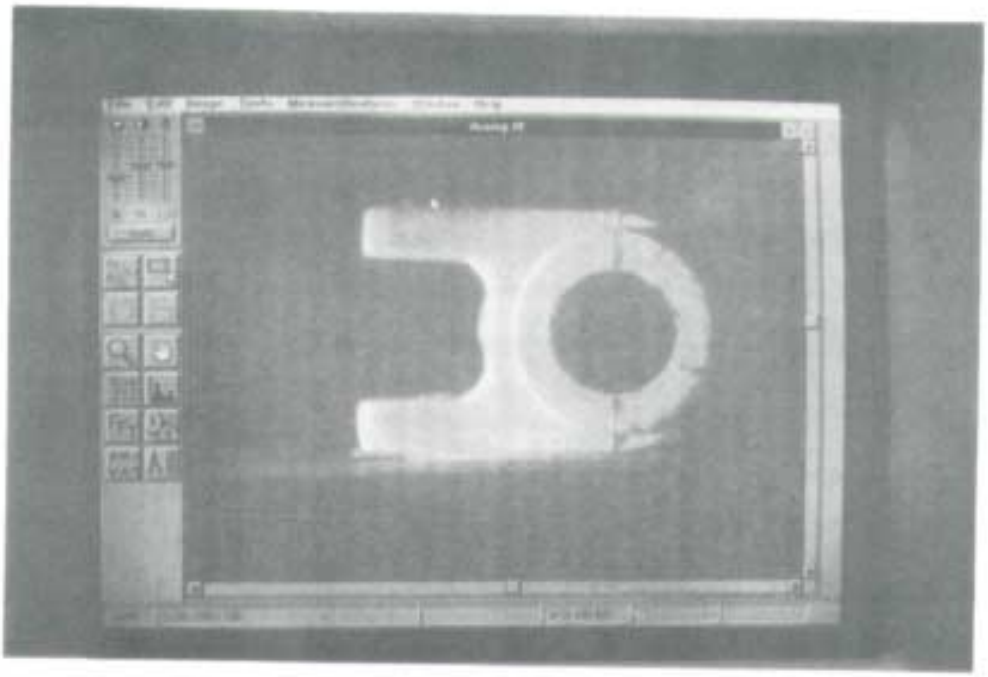

Figure 3. Green part in furnace.

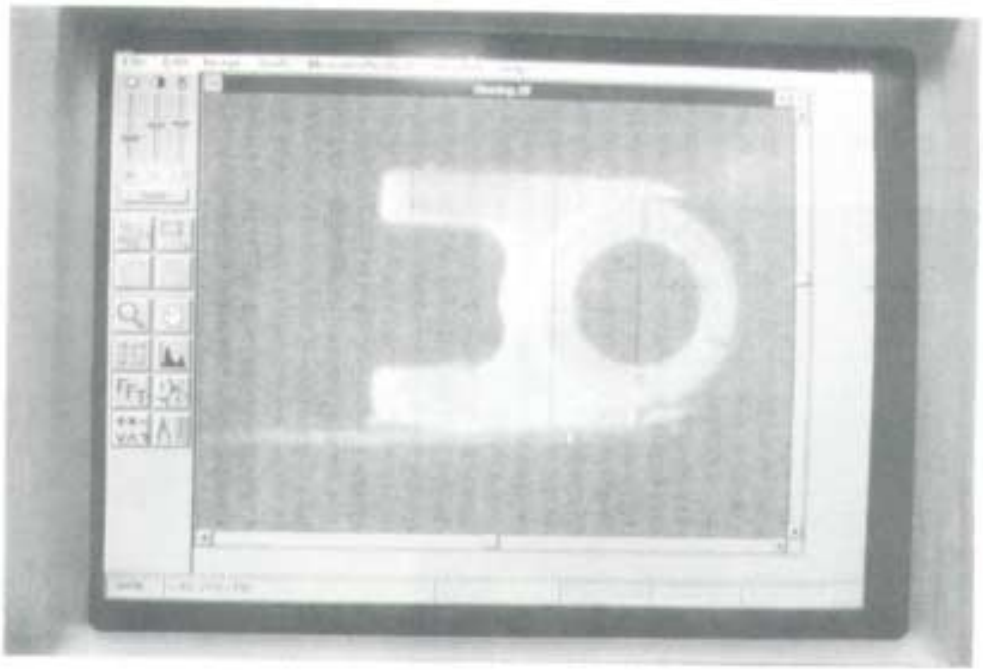

Figure 4. Green part in furnace.

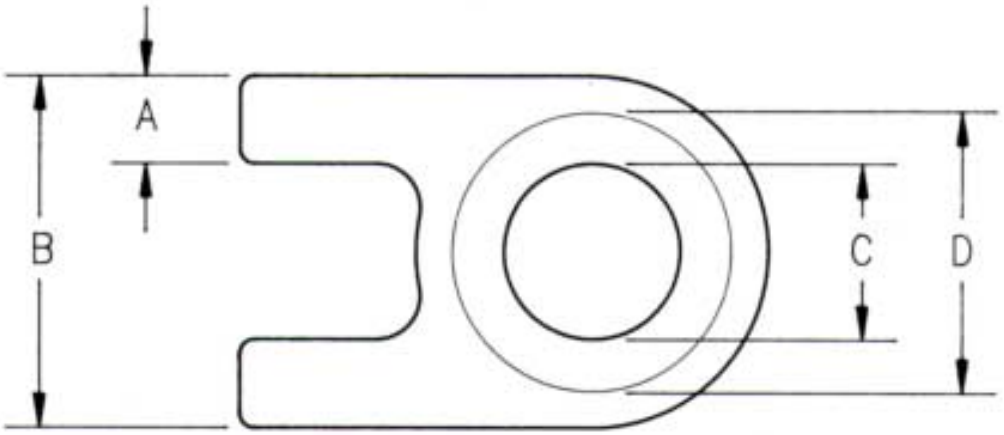

Figure 5. Dimensions of part measured.

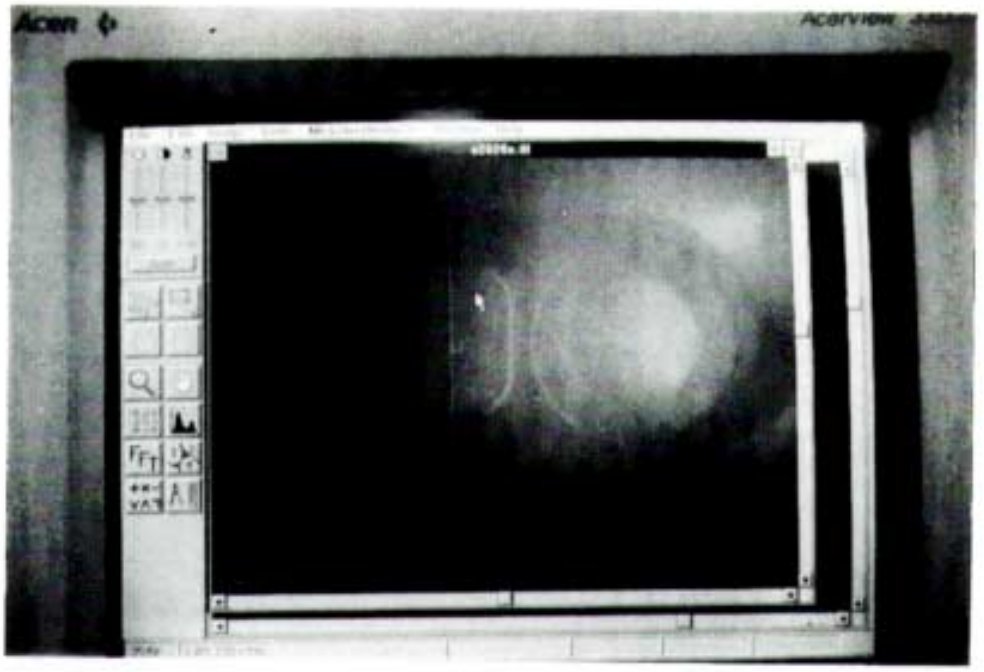

Figure 6. Part at end of presinter soak at 1000°C. Note sharp edge of Circle D.

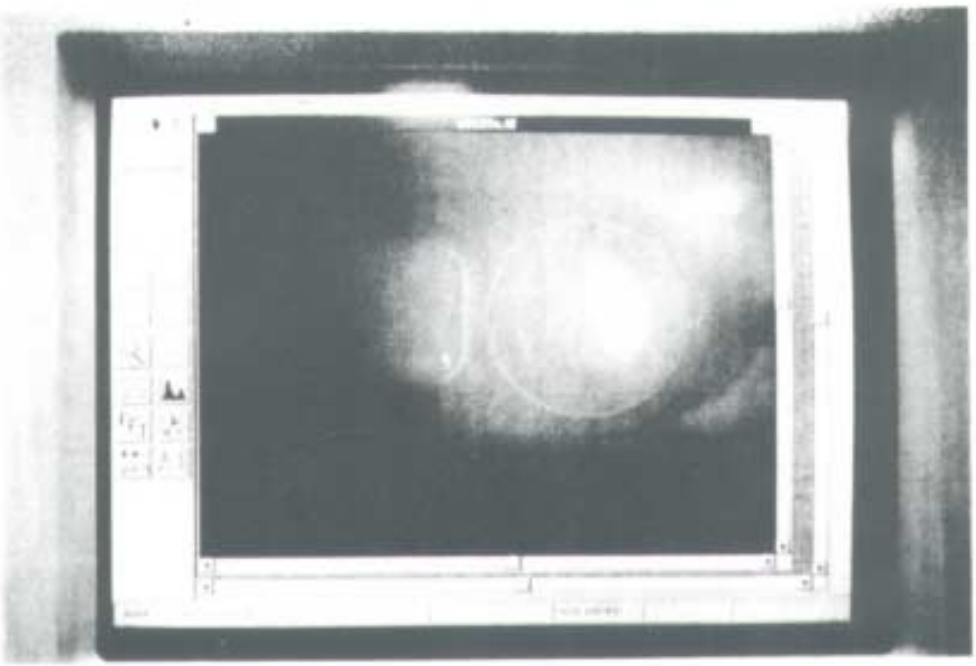

Figure 7. Part at end of presinter soak, with computer defined diameter on Circle D.

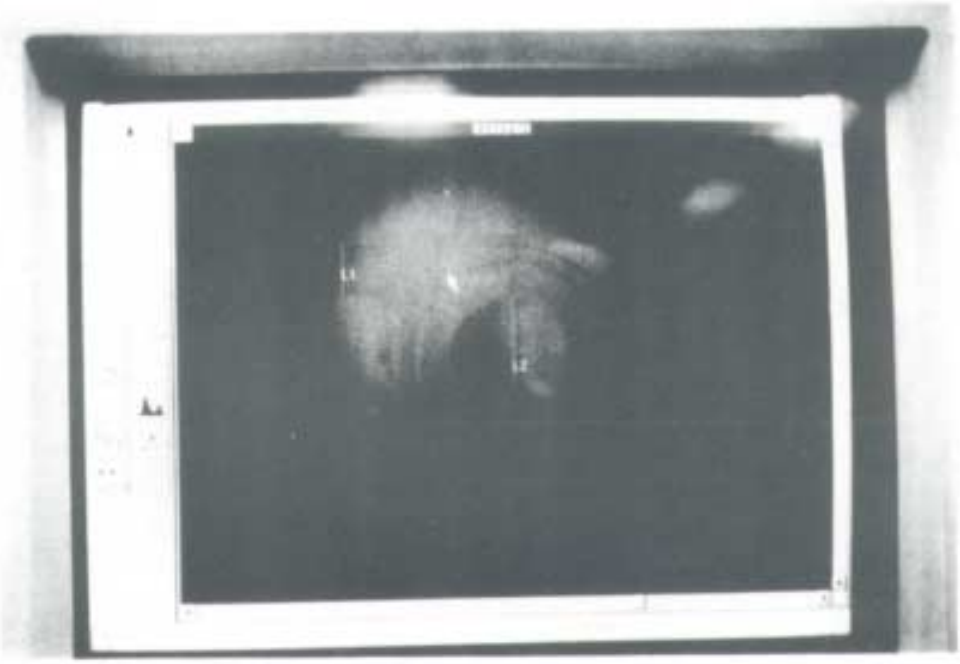

Figure 8. Sixty minutes into 1200°C sinter.

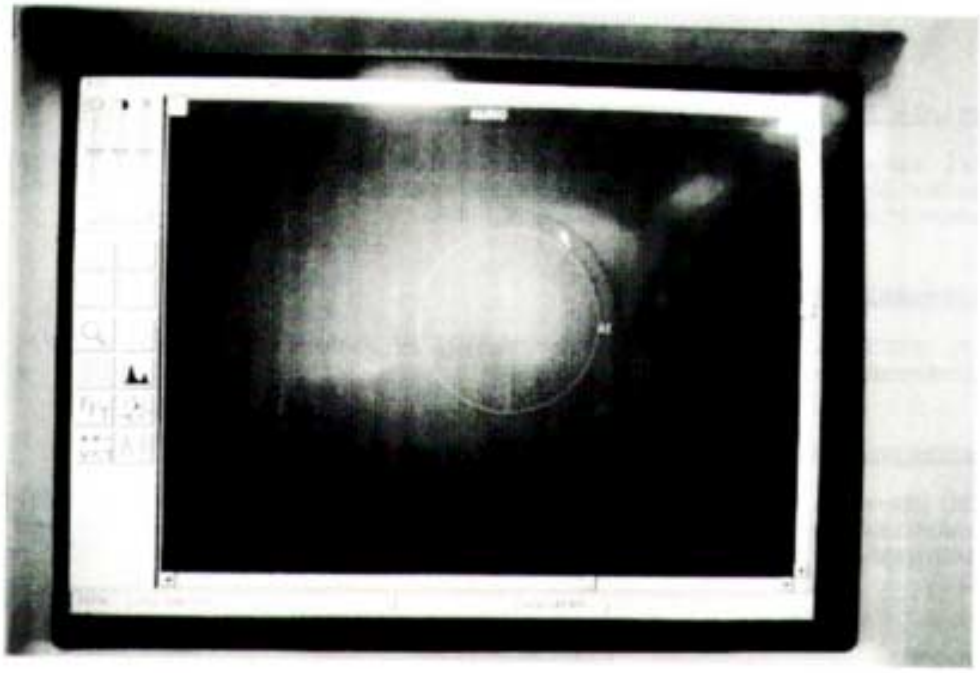

Figure 9. Ninety minutes into 1200°C sinter.

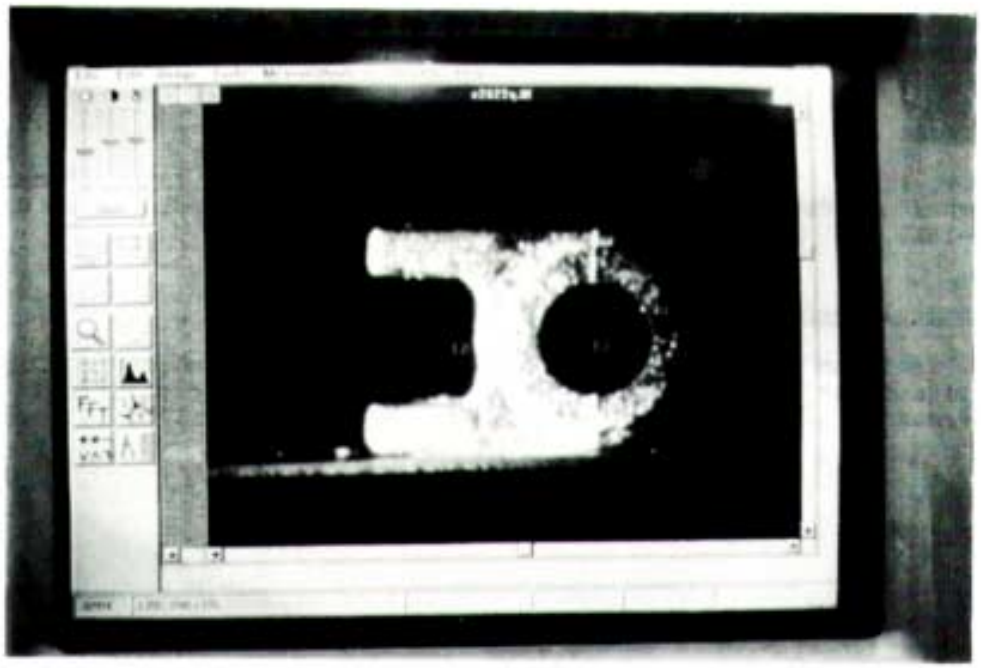

Figure 10. Part in furnace after cooling.

REFERENCES

1) Article in Book/Conference Proceedings

Karl Hens, "Tooling and Instrumentation for Quality", *1993 International Powder Injection Molding Symposium*, October 4-6, 1993, Metal Powder Industries Federation, Princeton, NJ.

2) Technical Report

H.T. Yolkem, "Intelligent Processing of Materials", *NIST 1992*, Garthersbury, MD.

3) Technical Report

N.M. Werely, T.F. Zahrah and F.H. Charron, "Intelligent Control of Consolidation Processes", *Journal of Materials Engineering and Performance*, Vol. 2, No. 5, Materials Park, OH, October 1993, pp. 671-682.

4) Journal

P.A. Parish and W. G. Barker, "The Basics of Intelligent Processing of Materials, *JOM*, July 1990, pp. 14-16.

5) Article in Book/Conference Proceedings

D.P. Duncavage and C.W.P. Finn, "Debinding and Sintering of Metal Injection Molded 316L Stainless Steel", "Powder Injection Molding, Advances in Powder Metallurgy & Particulate Materials", *Metal Powder Industries Federation*, Princeton, NJ, Vol. 5, 1993, pp. 91-103

6) Journal

C.W.P. Finn, "Vacuum Binder Removal and Collection", *International Journal of Powder Metallurgy*, Vol. 27, No. 2, APMI, Princeton, NJ, pp. 127-132.

7) Technical Report

D.W. Yoel and D.M. Garman, "Transparent Furnace Technology for Space Applications", *38th Congress of the International Astronautical Federation*, IAF-87-407, Brighton, UK, October 10-17, 1987.