

## Various Master Sintering Curve Concepts and its Applications

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### Abstract

The master sintering curve (MSC) is derived from densification data over a range of heating rates and temperatures. To improve the accuracy, several modifications were proposed: multi-phase MSC for solid state sintering with phase changes, MSC for liquid phase sintering, and MSC with consideration of grain growth. The developed MSC models were applied to several material systems such as molybdenum, stainless steels, and tungsten heavy alloys (WHA), in order to evaluate the effect of compaction pressure, phase change, grain growth, and composition on densification, to classify regions having different sintering mechanism, and to help engineer design, optimize, and monitor sintering cycles.

**Keywords:** master sintering curve (MSC), multi-phase sintering, densification, and grain growth

### 1. Introduction

The MSC is a simple and effective sinter model, used to predict the sintering densification of a porous body formed from powder. Sinter models, using the MSC approach, have been determined for powder ceramics as well as for powder metals [1, 2]. These studies have shown that the MSC is a powerful tool in helping determine the dominant sintering mechanisms through diffusional activation energy analysis, as well as providing a predictive model for estimating density as a function of thermal cycle (time and temperature profile). In this study, several modifications of MSC are proposed in order to evaluate the effect of compaction pressure, phase change, grain growth, and compositions on sintering densification.

### 2. MSC Modeling and Results

The conventional MSC as defined by Johnson [1], links the work-of-sintering  $\Theta$  [4],

$$\Theta = \int_0^t \frac{1}{T} \exp\left(-\frac{Q}{RT}\right) dt \quad (1)$$

to the relative sinter density,  $\rho$  at time  $t$  during the thermal cycle, starting at  $t = 0$ . The units of time and temperature used for calculation are second and Kelvin, respectively. The activation energy  $Q$  for the sintering system is either determined through minimizing the error between the experimental data and the model, or it is assigned a value based on known diffusional activation energy for the system [1,3]. It has been shown [2,3] that a sigmoid function

provides a good fit between the relative sinter density and the natural logarithm of the work-of-sintering,  $\ln \Theta$ . The sigmoid equation used to define the MSC is

$$\Psi = \frac{\rho - \rho_0}{1 - \rho_0} = \frac{1}{1 + \exp\left[-\frac{\ln \Theta - a}{b}\right]} \quad (2)$$

where  $\Psi$  is the densification parameter,  $\rho_0$  is the relative density at the start of the sintering experiment, and  $a$  and  $b$  are constants defining the curve. An alternative form of Eq. (2) is

$$\ln \Phi = \ln \left[ \frac{\rho - \rho_0}{1 - \rho} \right] = n (\ln \Theta - \ln \Theta_{ref}) \quad (3)$$

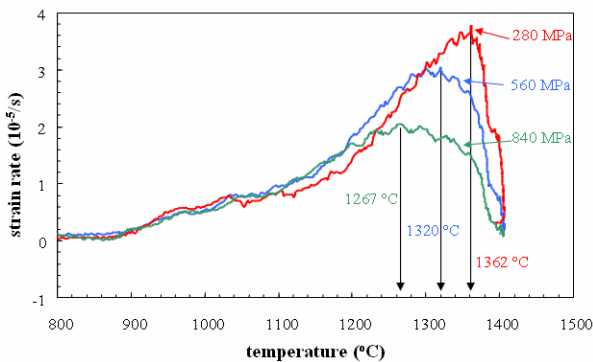
where  $\Phi$  is called the densification ratio.

### 3. Results and Discussion

The MSC is characterized by a series of constant heating rate or isothermal hold sintering experiments [1]. In this study, three constant heating rate experiments were conducted in a vertical pushrod dilatometer. The samples were prepared both by die compaction and powder injection molding with molybdenum, 17-4 PH stainless steel, 316 L stainless steel with boron, and WHAs.

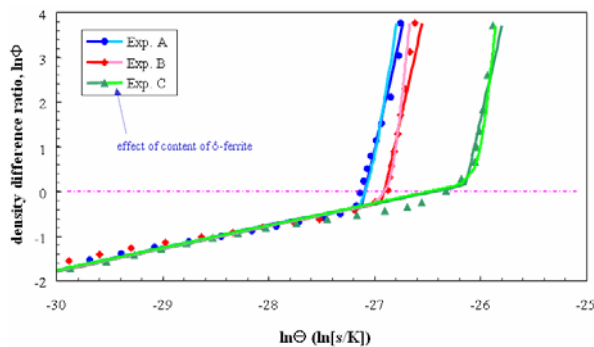
Fig. 1 shows the shrinkage behavior of the molybdenum at different compaction pressures. (The total shrinkage and the shrinkage rate decreases with pressure because of the starting green density increment) The total shrinkage and

the shrinkage rate decrease as the compaction pressure is increased due to the higher green density achieved at higher compaction pressures. However, the temperature at maximum shrinkage rate also decreases as the compaction pressure increases (with pressure). This indicates that even though the activation energy for the MSC increases as compaction pressure is increased; the samples pressed at high pressures (achieve) sinter to a higher density at lower temperatures than samples pressed at low pressures. This clearly indicates the importance of compaction pressure on the sintering of molybdenum [4].



**Fig. 1. Densification behavior with effect of compaction pressure for molybdenum.**

A new two-phase MSC model for densification of 17-4PH stainless steel, with consideration of  $\delta$ -ferrite content, is developed. When comparing experimental dilatometry results with the predictive model, it is clear that the two-phase MSC gives a closer prediction across the entire thermal cycle, as shown in Fig. 2 [5].

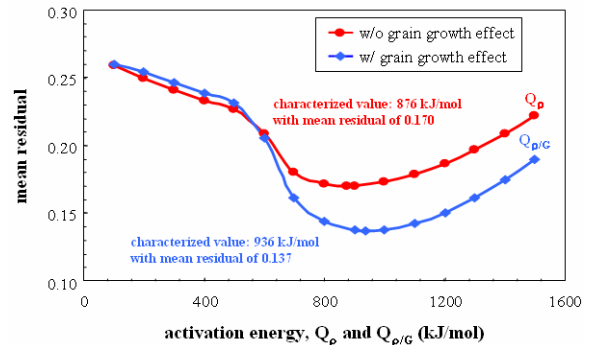


**Fig. 2. MSC with consideration of  $\delta$ -ferrite amount for 17-4 PH stainless steel.**

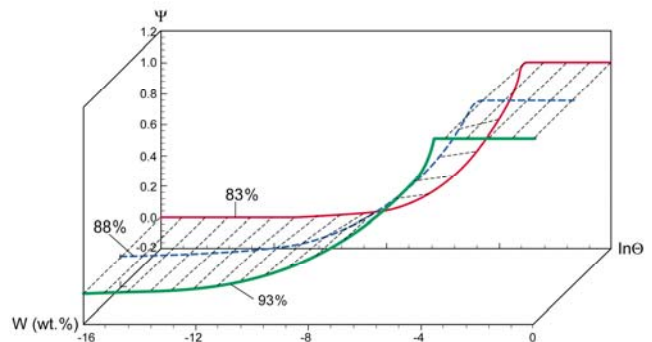
Fig. 3 shows the calculated apparent activation energy with consideration of grain growth has a smaller mean residual error than that without consideration, which means that a MSC of densification behavior that includes grain growth can predict sintering densification more accurately [6].

Fig. 4 shows the master sintering surface (MSS), constructed from a series of MSCs for WHAs with varying tungsten content. From this plot, the density can be

predicted for any given 83 to 93 wt.% WHA system and sintering cycle. Such MSSs are very useful for determining optimum combinations of material compositions and sintering cycles [7].



**Fig. 3. Apparent activation energy with consideration of grain growth for 316L stainless steel.**



**Fig. 4. MSC surface with consideration of composition for tungsten heavy alloys.**

#### 4. Summary

Several MSCs are proposed and demonstrated in order to evaluate effect of compaction pressure, phase change, grain growth, and compositions on densification. These MSCs showcase the accurate predictive capabilities for final density of this sinter model.

#### 5. References

1. H. Su and D. L. Johnson, *J. Am. Ceram. Soc.*, **79**[12], p.3211 (1006).
2. M-H. Teng, Y-C. Lai, and Y-T. Chen, *West. Pac. Earth Sc.*, **2**[2], p.171 (2002).
3. D. C. Blaine, J. D. Gurosik, S. J. Park, D. Heaney, and R. M. German, *Met. Mat. Trans.*, **37A**, p.715 (2006).
4. P. Garg, S. J. Park, and R. M. German, *Int. J. Refract. Hard Met.*, accepted (2006).
5. D. Blaine, S. J. Park, and R. German, *Sintering '05*, Grenoble, France, 2005.
6. R. Bollina, Ph.D. thesis, Penn State University (2005).
7. S. J. Park, J. M. Martin, J. F. Guo, J. L. Johnson and R. M. German, *Met. Mat. Trans.*, submitted (2006).