THE CONSOLIDATION BEHAVIOR

OF METAL COATED FIBERS

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Abstract

An experimental study has been conducted on the consolidation of randomly packed arrays of titanium alloy coated silicon carbide fibers. The densification rate was measured in situ using an eddy current sensor and compared to the predictions of a model. The model addressed densification by plasticity, power law creep and diffusion accommodated grain sliding. It utilized the results of detailed finite element analyses on the blunting of fiber reinforced contacts and analytical models for the closure of isolated voids. The results reveal that in physical vapor deposited Ti-6Al-4V coated fibers, diffusion accommodated grain sliding is the dominant densification mechanism. When time and temperature dependent microstructure relations were incorporated into the model, the simulated predictions were found to agree well with the HIP consolidation experiment.

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Introduction

The relatively high specific stiffness, strength, and creep resistance of metal matrix composites (MMC’s) consisting of titanium or nickel alloy matrices reinforced with continuous silicon carbide monofilament fibers have attracted considerable interest [1]. In one synthesis approach, the matrix alloy is evaporated (using physical vapor deposition techniques such as sputtering or electron beam evaporation) and directly deposited onto the fibers [1-4]. This results in matrix coated fibers like those shown in Fig. 1. During deposition, the fiber temperature remains low resulting in the formation of a nanocrystalline matrix microstructure [4].

After the fibers have been coated, they are packed into a die and consolidated to near net shape using a hot isostatic press (HIP). The consolidation process must result in complete densification, (see Fig. 2), while simultaneously avoiding mechanical or chemical damage to the fiber or its interfacial coating. Such damage results in a reduction of the composite’s properties [5]. Since many variables affect these time varying pressure/temperature dependent mechanisms, it has been useful for other synthesis routes (e.g. the consolidation of plasma spray deposited monotapes [6,7]) to develop models for the consolidation process. These models could then be used to find optimal process schedules designed to attain full density while minimizing fiber bending/fracture and reaction zone thickness [8,9]. Models of this type describe densification, fiber damage, and interfacial reaction kinetics as functions of fabrication pressure, temperature, and time when given, as inputs, the initial packing geometry and the matrix/fiber constitutive properties.

![Figure 1. Initial packing of a bundle of Sigma 1240 fibers sputter coated with a Ti-6Al-4V matrix (as-received condition).](image-url)
At first sight, the densification modeling problem encountered during the consolidation of metal coated fibers appears straightforward. A micromechanical analysis of contact deformation combined with the number of fiber-fiber contacts during densification could be used to predict early densification [10] while the analysis of isolated voids using suitable potentials [11] provides the required results when the density is greater than 90%. In reality, the problem must also address the evolution of contacts in a random packing, the lack of a geometrically accurate final stage potential, and account for the unique mechanical properties resulting from the PVD process. The PVD matrices are initially nanocrystalline and recent experimental studies with sputtered Ti-6Al-4V have shown that this leads to an enhanced superplastic effect when the temperature is within the near equal $\alpha + \beta$ phase region (i.e. 760 - 900°C) [4]. During consolidation, rapid grain coarsening occurs at elevated temperatures, and the mechanical behavior of the matrix then becomes a sensitive function of the process conditions [4].

In this work a model has been developed for the HIP consolidation of silicon carbide fibers which have been sputter coated with a Ti-6Al-4V matrix. It incorporates the mechanics of consolidation, the dominant deformation mechanisms of superplastic titanium, and the time/temperature microstructural evolution. Using a hot isostatic press containing an eddy current sensor to monitor sample dimensions, the densification response of a cylindrical specimen was measured in situ. The model was then used to simulate the HIP cycle and the resulting densification response was compared with the experiment.
Experimental Procedure

The tungsten cored Sigma 1240 fiber was used in the consolidation study. The Sigma 1240 fiber has a nominal diameter of 100 µm and a carbon-TiB₂ duplex coating [12]. The Ti-6Al-4V matrix was deposited on the fibers via a sputtering process at 3M Metal Matrix Composite Center (Mendota Heights, MN), and resulted in a roughly elliptical matrix coating, Fig. 1, with a nanometer scale grain size (~30-100 nm) [4]. Bundles of approximately 4000 matrix coated fibers were packed in a 16 mm diameter cylindrical canister [13]. The canister was placed within an encircling eddy current sensor [13-16], and HIPped using the process cycle shown in Fig. 3 [13].

The temperature was raised to 840°C after an intermediate hold at approximately 200°C during which the pressure chamber was purged. The temperature ramp rate was 10°C/min. Once the temperature setpoint was reached, the system was held for 15 minutes to allow thermal equilibrium to be established within both the specimen, eddy current sensor, and pressure chamber. The vessel was then pressurized to a low pressure setpoint of 20 MPa. Such a “gentle” HIP cycle has been shown to minimize the fiber damage that occurs as a result of fiber crossovers in a random bundle [17]. After a 30 minute hold, the pressure was increased to 100 MPa and held for an additional 60 minutes. The pressurization rate was approximately 5 MPa/min for chamber pressures less than 7 MPa and 1 MPa/min for pressures up to 100 MPa.

After consolidation, the specimen’s final diameter was measured. Cross-sections of the sample were then mounted in epoxy and polished. Upon examination in a scanning electron microscope, the specimen was found to be fully dense. Using an image analysis software package, the volume fraction of fiber was determined to be 0.494 ± 0.015.

![Figure 3. HIP cycle profiles showing the variation of density, temperature, and pressure with time.](image-url)
Throughout the cycle, the sensor measured the sample diameter, $d_r$, at one minute intervals from which the density, $D_r$, was computed after correcting for the thermal expansion contribution of the sample and sensor [13]. The instantaneous density, $D_r$, was related to that measured after consolidation by

$$D_r = \frac{\pi/4d_f^2 - a_w}{\pi/4d_i^2 - a_w}$$

(1)

where $D_f$ is the final density, $d_f$ is the final diameter, and $a_w$ is the area of the canister wall which is assumed to be constant. Using equation (1), the densification response of the specimen, shown in Fig. 3, was calculated for the entire cycle.

**Densification Mechanisms**

Three mechanisms for densification of PVD matrix coated fiber bundles have been identified: plasticity, diffusion accommodated grain sliding, and power law creep [18]. In keeping with past attempts to model the densification of powders and plasma sprayed foils [6,19,20], the densification process was modeled in two stages. As shown in Fig. 4, Stage I consists of the deformation (or blunting) of individual contacts, and Stage II involves the collapse of isolated voids. During Stage I the contacts are considered to be isolated from each other while in Stage II the voids do not interact. The transition between Stage I and II occurs when the relative density reaches 90%.

For a power law creeping material, the time dependent deformation mechanisms can be expressed by a relation of the form

$$\frac{\sigma}{\sigma_o} = F_c \left(\frac{\dot{\varepsilon}}{\dot{\varepsilon}_o}\right)^{1/n}$$

(2)

where $n$ is the creep exponent, $\sigma$ is the stress, $\dot{\varepsilon}$ is the strain rate, $\sigma_o$ is the reference stress, $\dot{\varepsilon}_o$ is the reference strain rate, and $F_c$ is a creep flow coefficient. Both reference values are temperature dependent material properties. In Stage I, the stress and strain rate are defined at the contact [18] while in Stage II they are the applied stress and macroscopic strain rate [21].

**Diffusion Accommodated Grain Sliding**

Diffusion accommodated grain sliding (DAGS) has been shown to be the dominant deformation mechanism in material produced via a PVD method due to the nanocrystalline microstructure [4]. The combination of DAGS and power law creep (PLC) has been successfully used to model the superplastic behavior of several materials [4,22]. Ashby and Verral [22] have developed an expression for the strain rate of a material due to DAGS

$$\dot{\varepsilon}_{DAGS} = \frac{100\Omega D_v}{kTd^2} \left(\frac{0.72\Gamma}{d}\right) \left(1 - \frac{3.38\delta D_g}{dD_v}\right)$$

(3)
where $\sigma_c$ is the contact stress, $T$ is the temperature, $d$ is the grain size, $\Gamma$ is the grain boundary energy, $\delta$ is the grain boundary width, $\Omega$ is atomic volume, $D_v$ and $D_{gb}$ are vacancy and grain boundary diffusion respectively, and $k$ is Boltzmann's constant.

**Power Law Creep.**

The second mechanism used in modeling superplastic behavior is power law creep. The strain rate of a power law creeping material under uniaxial loading may be described by

$$\dot{\varepsilon}_{PLC} = A \left( \frac{\mu b}{kT} \right)^n D_v$$

where $\mu$ is the temperature dependent shear modulus, $b$ is the burger's vector, $A$ is a material property, and $n$ is the power law exponent, also a material property.

![Diagram](image-url)

**Figure 4.** Model geometries for a) the isolated contacts in Stage I and b) the individual voids of Stage II.
Plasticity

Plasticity is taken to be a time independent mechanism. It is possible to obtain the case of perfectly plastic behavior by letting the power law exponent, $n$, approach infinity. Taking the limit of equation (2) as $n$ approaches infinity results in

$$\sigma = F_p \sigma_y$$

(5)

where $\sigma_y$ becomes the uniaxial yield strength, $\sigma_y$, of the matrix material.

Plasticity occurs when the stress equals or exceeds the matrix yield strength multiplied by the flow coefficient, $F_p$. For this mechanism, the flow coefficient represents the resistance of the matrix to plastic flow. It has been determined to be geometry dependent (varying in both Stage I and Stage II) and a function of both density and volume fraction of fiber [10,21,23].

Stage I

All of the Stage I micromechanics involve the deformation of individual contacts (Fig. 4a). It is possible to model the deformation of a contact with two coefficients, $F$ and $c$ [10,23]. The coefficients, $F_c$ and $F_p$, in equations (2) and (5) represent the materials resistance to creep and plastic flow, respectively.

The manner in which the contact length increases with the amount of contact deformation is reflected within the coefficient, $c$. During Stage I, the amount of deformation is simply the normal displacement, $h$, that each contact undergoes during densification. The relationship between $c$ and $h$ can be defined as

$$h = \frac{1}{2c^2} \frac{a^2}{r_o}$$

(6)

where $a$ is half of the total contact length, $h$ is the normal displacement, $r_o$ is the initial radius of the matrix coating, and $c$ is the contact area coefficient.

For a creeping material, Gampala et al [23] have determined that the coefficients $F_c$ and $c$ are dependent upon both the creep exponent, $n$, and the fiber volume fraction, $\nu_f$. Throughout Stage I, the results for their relationships are used in calculating the densification rate of the time dependent mechanisms. For densification by plasticity, we use the results obtained by Wadley et al [10] who determined the effect the volume fraction of fiber has on both the plastic flow coefficient, $F_p$, and the contact area coefficient, $c$. With these results, it is possible to determine the pressure required to cause densification by plasticity.

Stage II

In Stage II, the representative volume elements are composed of individual cusp-shaped voids as shown in Fig. 4b. Voids of this shape have been experimentally identified in the consolidation of matrix coated fibers [13]. Qian et al [21] have used a potential method to
model the densification of various types of cusp-shaped voids by introducing a shape factor. Here, we use the results of their work in modeling Stage II.

Qian et al do not account for the effect of the fiber on the densification process. Although at high densities ($D > 97\%$) the fiber has little affect on the densification rate, the effect is significant at densities of 90% and greater [23]. We incorporate the effect of the fiber, by “calibrating” the Stage II model using the results of Gampala et al’s unreinforced and fiber reinforced cases [23]. The calibration introduces a restraint factor into the model of Qian et al. At high densities, the restraint factor is reduced to unity using an exponential function, thus recovering Qian et al’s original result. Details on the manner in which the restraint factor was determined and incorporated into the model may be found in reference [18].

**Implementation of the Model**

The material system used in the experimental consolidation study was a Ti-6Al-4V matrix and a Sigma 1240 (SiC) fiber. The material properties of Ti-6Al-4V as published by Warren et al [4] are given in Table I. In order to effectively simulate the densification process, several factors concerning the matrix must be addressed. First, Ti-6Al-4V is a two phase material at 840°C. It is possible to account for the two phases by imposing an isostrain/isostrain-rate condition [4]. The stresses are then partitioned into each phase according to the rule of mixtures. By doing so, the contribution of each mechanism to the overall densification rate can be determined.

The second consideration is the extremely small grain size of the PVD deposited matrix. As may be seen in equation (3), the DAGS strain rate is a function of $1/D^2$ and so is a very sensitive function of the grain size. In order to simulate the consolidation behavior of the coated fibers accurately, the evolution of the grain size must be considered. In their study of the deformation
of PVD Ti-6Al-4V, Warren et al [4] determined the grain growth kinetics for this material at 840°C to be

\[ d = 0.20 + 0.23t^{0.20} \]  \hspace{1cm} (7)

where \( d \) is the average grain size in microns and \( t \) is the time in seconds.

With the above material properties and models it is possible to simulate the HIP process. Densification due to plasticity, the rate independent mechanism, is calculated for both Stage I and II by directly solving equation (5), given the appropriate relationship for \( F \) as a function of density. The densification rates of the time dependent mechanisms are computed at each density and summed resulting in the overall densification rate. Each mechanism is assumed to act independently. More details on the implementation of the model are given in reference [18].

**HIP Cycle Simulations**

Simulations of the HIP experiment have been conducted using the consolidation model [18]. As an input into the model, the volume fraction of fiber determined by the image analysis, 0.494, was used. The simulation used the density (0.720) at which the pressure ramp began as the initial condition. The pressure was ramped to the appropriate setpoint at the same pressurization rate as in the experiments. During the simulation, the time dependent grain growth kinetics determined by Warren et al [4] were used to calculate the average grain size and determine the contribution of the grain size dependent mechanisms. The results of the process simulations along with the experimentally measured data are shown in Fig. 5.

![Figure 5. Experimental densification profile and model simulation.](image-url)
Discussion and Summary

During Stage I, very good agreement has been observed between the model and the experiment. In this stage, the densification rate is rapid due, in part, to the low number of contacts and the small contact area resulting in a high stress at each contact. Additionally, the small grain size of the PVD matrix increases the contribution of DAGS making it the dominant densification mechanism. This results in the attainment of a high relative density (~94%) in less than one hour at a relatively low pressure of 20 MPa. In Stage II, the model slightly underestimated the density. This appears to have arisen from the approximate way in which the fiber’s effect was integrated into the Stage II modeling. Greater analysis of this area is required to more accurately determine the effect of the fiber.

In summary, a cylindrical specimen containing a randomly packed bundle of metal matrix coated fibers has been HIPped and the densification response measured using an eddy current sensor. A model was then developed and used to simulate the HIP process. The resulting densification profiles showed very good agreement over the entire range of density.

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