Densification Modeling of Titanium Alloy Powder during Hot Isostatic Pressing

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Abstract:
Densification model of titanium alloy (Ti–6Al–4V) are investigated during hot isostatic pressing (Hip). Experimental data was obtained at various pressures and temperatures during hot isostatic pressing (Hip). Experimental data are compared with the finite element calculations by using the hybrid model and Abouaf model, respectively. The results show that the finite element calculation results by the hybrid model are in agreement with the experimental data for densification behaviour of the titanium alloy powder under Hip; however, the finite element calculation results by using the Abouaf model are over the experimental data. In addition, in order to obtaining relative density distributions of porous body, the statistical relationships during Poisson’s ratio, Rockwell hardness and the relative density of porous body were formulated, the results show that the statistical relationship between Poisson’s ratio and the relative density of porous body is essential to construct such a constitutive model; the statistical relationship between Rockwell hardness and the relative density of porous body is essential to obtain relative density distributions of porous body.

Keywords: Constitutive model, Densification, Finite element analysis, Hot isostatic pressing, Titanium alloy.

1. Introduction
Owing to their high specific strength, excellent fracture toughness, good creep and corrosion resistance, titanium alloys have been used in many fields. However, high cost of raw materials, alloys, and final products severely restricts the wide applications of titanium alloys. Development of low cost titanium alloys through adding cheap elements is an effective method for reducing costs. Another way is near net shape forming, such as powder metallurgy (PM) [1]. Hip is one of the most widely used PM in the manufacturing process of high strength materials [2]. For geometry control and good mechanical properties of a final part, it is necessary to predict the density distribution of the Hipped part [3]. Numerical simulations using finite element analysis are very useful in studying the density distribution and the deformation shape of a powder compact. The finite element modelling requires a constitutive

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model to represent the deformation behaviors of powder. Recently, several theoretical models [4-10] have been proposed to predict densification during Hip of powders on the basis of creep and plastic deformation mechanisms during the compaction of monosized spherical powder particles. The development of these models relies extensively on experiments by Swinkels et al. [4], who examined the hot isostatic pressing behavior on lead, tin, and polymethylmethacralate powders. In particular, the analysis of Arzt et al. [5] incorporated plastic yielding, power-law creep, and diffusional densification in order to predict densification of equisized, spherical powders as a function of Hip parameters. Subsequently, Helle et al. [6] have extended this model such that densification rates are predicted under a wider range of densification mechanisms. All analyses [5-7] provide the basis for constructing Hip maps. Carroll [8] has analyzed the problem of rapid density increases associated with an initial rapid change in pressure (30 seconds rise time). In addition, Nair and Tien [9] have proposed an analysis of Hip for the compaction of powders of unequal size. However, the Hip maps including plastic yielding, power-law creep, and diffusional densification is so complicated that it is not convenient for numerical simulations using finite element analysis. More recently, Song et al. [10] and Kim and Kim [11] studied the power-law creep densification of metal powder under constant pressure during hot pressing by using the constitutive models proposed by McMeeking and co-workers [12,13]. Shaik and Milligan [14] investigated the densification of metal powder under various loading rates and pressure during hot pressing by using the creep model of Arzt et al. [5]. However, they did not consider thermo-plastic deformation and densification of metal powder with low relative density.

The present paper investigates the densification model of titanium alloy (Ti–6Al–4V) powder during Hip. We consider Hip as a thermo-plastic or creep densification. Experimental data were obtained for titanium alloy powder at various pressures and temperatures during Hip. The parameters of Abouaf [15] model and the hybrid model incorporating both Fleck model and modified Gurson model were determined by uniaxial compression and uniaxial compression creep on samples which have been hot isostatically pressed to the relative density range of 70%-100%. The constitutive models of Fleck et al. [16] and the modified Gurson [17, 18 and 19] were employed for the finite element calculations to predict thermo-plastic densification behavior of porous titanium alloy during Hip. We also used the constitutive models of Abouaf and co-workers [15] for creep densification of titanium alloy powder under Hip. Experimental data were compared with finite element calculations for deformed shape of the container and relative density distributions during Hipping.

2. Experimental

Argon gas atomized spherical titanium alloy powder (Ti–6Al–4V, TLK Technik, Germany) with a theoretical density of 4.43gcm$^{-3}$ and a melting point of 1727 was used in this work [20]. The powder consisted of spherical particles with a mean diameter of 150μm.

2.1 Hot Isostatic Pressing

The powder was loaded into low carbon steel containers (35mm height, 28mm diameter, 1.5mm wall thickness) which were sealed via electron-beam welding. The canned powders were consolidated from an initial relative density of 65% to various relative densities in a series of interrupted Hip experiments. Each experiment was terminated at a predetermined time to produce final relative densities ranging from 78 to 100%, as shown in Fig.1.
The relative densities of the powder compacts were determined by Archimede's laws. Density distributions of a powder compact can be obtained indirectly from the relationship between hardness and relative density. Hardness of titanium alloy powder compacts was measured by a Rockwell(type B) tester with a 1.58mm hardened steel ball indenter by applying 100kg force on the sample for 30s in this work.

According to actual need, a sample was cut by a wire cutting machine. Then, the sample was annealed at 600°C for 2h to remove possible residual stress. Some key points were measured for each sample to obtain the relationship between hardness and relative density.

2.2. Uniaxial Compression of Titanium Alloy Powder Compact

To obtain uniaxial stress–plastic strain responses of fully dense titanium alloy powder compacts at various high temperatures and with various strain rates, Gleeble1500 tester was used. To reduce the friction between the sample and platens during compression, refined graphite dust was filled into circular groove located in two ends of a sample. The temperature was raised at a rate of 10°Cmin⁻¹ up to test conditions. Uniaxial compression tests were done at 750, 850, 950 and 1050°C, and respectively with various strain rates (0.001s⁻¹, 0.01s⁻¹, 0.1s⁻¹), in an argon atmosphere.

2.3. Uniaxial Creep of Solid Titanium Alloy

The creep constants in power-law creep response of solid titanium alloy were obtained from uniaxial compression creep tests of fully dense samples under high temperature. The fully dense titanium alloy samples were produced by Hipping of titanium alloy powder in this work. A dead-weight-creep-rupture tester with a vacuum/environmental furnace was used for uniaxial compression creep of solid titanium alloy. Uniaxial compression tests were carried out in a stress range from 280 to 550MPa at 500°C, and in a stress range from 90 to 330MPa at 600°C. Cylindrical specimens measuring 8mm height ×10mm diameter EDM-machined from compacts were used for uniaxial compression creep test and uniaxial compression test. Uniaxial creep data (at 750, 850, and 950°C) obtained by Kim and Yang [21] were used.
3. Analysis
3.1 Constitutive Equations

The strain rate tensor for elastic–plastic response of a porous material may be written as:
\[ \dot{\varepsilon}_{ij} = \dot{\varepsilon}_{ij}^{e} + \dot{\varepsilon}_{ij}^{p} \]  
(1)

where \( \dot{\varepsilon}_{ij}^{e} \) and \( \dot{\varepsilon}_{ij}^{p} \) are the elastic and plastic strain tensors, respectively. The stress tensor \( \sigma_{ij} \) may be written from Hooke’s law. Thus,  
\[ \sigma_{ij} = D_{ijkl}^{e} \varepsilon_{kl}^{e} \]  
(2)

where \( D_{ijkl}^{e} \) is a fourth-order elastic modulus tensor.

The plastic strain rate tensor \( \dot{\varepsilon}_{ij}^{p} \) for a porous material may be written as:
\[ \dot{\varepsilon}_{ij}^{p} = \lambda \frac{\partial \phi}{\partial \sigma_{ij}} \]  
(3)

where \( \phi \) is the yield function for a porous material and \( \lambda \) is a positive scalar.

Assuming that a matrix material is incompressible, the rate of relative density \( \dot{D} \) can be written as:
\[ \dot{D} = -D_{kk} \dot{\varepsilon}_{kk}^{p} \]  
(4)

We also assume that the plastic work done on a porous material is equal to that of the matrix material. Thus,  
\[ D \sigma_{m} \dot{\varepsilon}_{ij}^{p} = \sigma_{ij} \dot{\varepsilon}_{ij}^{p} \]  
(5)

where \( \sigma_{m} \) is the flow stress and \( \dot{\varepsilon}_{ij}^{p} \) is the effective plastic strain rate of the matrix material.

3.2. Plastic Yield Functions by Fleck et al. and Gurson

From plastic deformation of spherical particles with perfectly plastic behaviour, Fleck et al. [16] proposed a yield function for the plastic response of a porous material with low relative density (\( D<0.9 \)). Thus,  
\[ \phi(\sigma, D) = \left( \frac{\sqrt{3} p}{3 P_{y}} \right)^{2} + \left( \frac{5q}{18 P_{y}} + \frac{2}{3} \right)^{2} - 1 = 0, \]  
(6)

\[ P_{y} = 2.97 D^{2} \frac{(D-D_{0})}{(1-D_{0})} \sigma_{m} \]  
(7)

where \( p \) (\( = -\sigma_{kk}/3 \)) is the hydrostatic stress, \( q \) (\( = \sqrt{3S_{ij} S_{ij}/2} \)) is the effective stress, and \( P_{y} \) is the yield strength under hydrostatic compression.

Tvergaard [18, 19] modified the yield function of Gurson [17] for a porous material with high relative density (\( D>0.9 \)). Thus,  
\[ \phi(\sigma, D) = \left( \frac{q}{\sigma_{m}} \right)^{2} + 2q (1-D) \cosh \left( \frac{3g_{2} p}{2\sigma_{m}} \right) - \left( g_{1} (1-D) \right)^{2} - 1 = 0 \]  
(8)

where the parameters \( q_{1} \) and \( q_{2} \) were introduced by Tvergaard [18, 19]. When \( q_{1} = q_{2} = 1 \), Eq. (8) reduces to the yield function originally proposed by Gurson [17].

To compare with the experimental data, we used the yield function, Eq. (7), of Fleck...
et al. [16] for the low density range (D<0.9) and Eq. (8), i.e. the yield function of the modified Gurson [17, 18, 19] for the high density range (D>0.9) during Hip. We used the interpolation equation suggested by Fleck et al. [16] for the continuity of the constitutive models in the density range where the applied constitutive model differs [22]. Thus,

\[ \phi = \left( \frac{D_2 - D}{D_2 - D_1} \right) \phi_1 + \left( \frac{D - D_1}{D_2 - D_1} \right) \phi_2 = 0 \]  

(9)

where \( \phi_1 \) and \( \phi_2 \) are the yield functions by Fleck et al. [16] and that of the modified Gurson [17, 18, 19], respectively. In Eq. (9), \( D_1 \) and \( D_2 \) are relative densities at which the transition begins and ends, respectively. For instance, we used \( D_1=0.75 \) and \( D_2=0.9 \) in this work [23]. We also assumed that Hip is an isothermal process at a specified high temperature.

3.3. Power-Law Creep Model by Abouaf and Co-workers

The general form of power-law creep for a metal can be written as,

\[ \dot{\varepsilon} = \dot{\varepsilon}_0 \left( \frac{\sigma}{\sigma_0} \right)^n = A \sigma^n \]  

(10)

where \( \dot{\varepsilon}_0 \) and \( \sigma_0 \) are the reference strain rate and reference stress, respectively. \( A \) and \( n \) are Dorn’s constant and the creep exponent, respectively. The equivalent Misses stress \( \sigma_{eq} \) for a porous material can be written as,

\[ \sigma_{eq} = fI_1 + 3cJ_2 \]  

(11)

where \( I_1 \) and \( J_2 \) are the first stress invariant and the second deviatoric stress invariant, respectively. When \( c=1 \) and \( f=0 \), \( \sigma_{eq} \) in Eq. (11) reduces to the usual Misses stress. The parameters \( c \) and \( f \) in Eq. (11) can be determined from experiments as functions of relative density \( D \) [24, 25]. Thus,

\[ f(D) = \frac{1}{9} \exp \left( \frac{\dot{D}}{D} \right) \left( \frac{1}{A \cdot P} \right)^{2/(n+1)} \]  

(12)

\[ c(D) = s(D)^{-2n/(n+1)} - f(D) \]  

(13)

where \( P \) denotes the external pressure subjected to the container of a sample, \( \exp \left( \dot{D}/D \right) \) can be obtained from experimental data \( D = D(t) \) under Hip. Also, \( s(D) \) can be obtained from uniaxial compression creep data of porous powder compacts under high temperatures [24, 25].

Assuming that the viscoplastic work of a matrix material is the same as that of a porous material, the creep strain rate \( \dot{\varepsilon}_{ij} \) can be written as,

\[ \dot{\varepsilon}_{ij} = DA\sigma_{eq}^{n-1} \left[ \frac{3}{2} cS_{ij} + fI_1 \delta_{ij} \right] \]  

(14)

where \( S_{ij} \) is the deviatoric stress, \( \delta_{ij} = \partial I_1 / \partial S_{ij} \) is the Kronecker delta.
3.4. Evolution of the Density during Hip

Once strains are known, the evolution of the density can be calculated using the following mass conservation equation during the deformation of the porous material,

\[
D_0 l_1 l_2 l_3 = D (l_1 + \Delta l_1) (l_2 + \Delta l_2) (l_3 + \Delta l_3)
\]  

(15)

where \(D_0\) and \(D\) are the relative densities before and after deformation, and \(l_i\) and \(\Delta l_i\) are linear dimensions of a material element along the \(\chi_i\) axis before and after deformation. It follows from Eq. (15) that,

\[
D = D_0 e^{-(\varepsilon_{11} + \varepsilon_{22} + \varepsilon_{33})} = D_0 e^{-\varepsilon_v}
\]  

(16)

where \(\varepsilon_v\) is the volumetric strain.

4. Results and discussion

4.1. Optical Microscopy of Polished Cross-Section for Hipped Samples

Hipped compacts with various relative densities ranging from 78 to 100% after EDM-machining can be seen from Fig. 2. Polished cross-sections were prepared for the specimens consolidated to 78%, 83%, 89%, 92% and 100% of theoretical density (See Fig. 3). At 78%, the interparticle contacts in the specimen are most notably characterized by very little particle deformation. The specimen consolidated to 83% relative density clearly includes particles that were deformed by contact with neighboring particles. At 83%, noticeable density variations were observed, apparently related to granular or shearing flow within the compact. At 89% relative density, the porosity was much more uniformly distributed, but some dispersed local variations remained, especially at the corners. At 92%, the porosity was partially isolated and partially interconnected, and pore cross-sections were clearly defined by sharp points near interparticle contacts. Fig. 3 (b) show that the interparticle contacts in the specimen consolidated to 83% relative density are most notably characterized by very little particle deformation. The specimen consolidated to 89% relative density [Fig. 3(c) and Fig. 3(e)] clearly includes particles that were deformed by contact with neighboring particles, and the micrographs reveal several rather interesting features of the deforming and densifying powder compact. The smaller particles in large–small interparticle contacts deformed more severely than the larger particles. In many cases, as is illustrated by the small particle in the center of Fig. 3(c), the deformation of the smaller particle was much greater than the surrounding larger particles. The pervasive difference in the relative deformation of large and small particles suggests a compaction mechanism involving rigid body motion of large particles or groups of particles facilitated by preferential deformation of smaller particles.

The micrographs also show clear evidence that tensile stresses, sufficient in magnitude to break interparticle bonds, developed in the compact during consolidation. Fig. 3(c) and Fig. 3(e) clearly show two examples in which the particles were previously in contact but became separated during further compaction. In both examples, but most clearly in Fig. 4, prior compressive stresses were sufficient to deform the particles. Evidence of a ductile fracture surface indicates that a diffusion bond formed between the particles before sufficiently large tensile stresses developed fracturing the interparticle bond and separating the particles. The presence of tensile fractures within a compressively loaded powder compact is direct evidence of the cooperative movement of clusters of particles by deformation, rotation, and translation during densification.
Fig. 2. Samples with various relative densities

Fig. 3. Optical microscopy of samples with various relative densities
4.2 Uniaxial Response of Solid Titanium Alloy

Fig. 4 shows the uniaxial stress–plastic strain response of titanium alloy at various high temperatures. The data points were obtained from the experimental data of dense titanium alloy. The solid curves were obtained from Eq. (17) by fitting the experimental data. Thus,

\[
\sigma_m = \begin{cases} 
75.57 + 121.32 \ast (\varepsilon) ^ {0.3574} \\
20.71 + 97.72 \ast (\varepsilon) ^ {0.3962} \\
9.17 + 23.12 \ast (\varepsilon) ^ {0.3423} \\
2.20 + 14.11 \ast (\varepsilon) ^ {0.3165}
\end{cases}
\]  

(17)

4.3. Constitutive Model Parameters

According to the latter solution of \( f \) and \( c \), as shown in Baccino [26], from uniaxial compression creep of dense samples, uniaxial compression on porous samples, and interrupted Hip trials consisting in densification with identical pressure and variable step durations, \( f \) and \( c \) in Eq. (14) can be written as,

\[
f(D) = 0.27 \left( \frac{1-D}{D-0.65} \right) ^ {0.9}
\]  

(18)

\[
c(D) = 1 + 0.7 \left( \frac{1-D}{D-0.65} \right) ^ {0.95}
\]  

(19)

Fig. 5 shows the variation of the parameter \( f(D) \) with relative density \( D \) for titanium alloy powder compacts. The data points represent experimental data of titanium alloy powder under Hip. The solid curve was from Eq. (18) and the dashed curve from Kim and Yang [21]. The solid curve from Eq. (18) agrees very well with the experimental data, however, the dashed curve from Kim and Yang [21] overestimates experimental data.

The constants \( A \) and \( n \) in Eq. (14) were determined from uniaxial compression creep. Fig. 6 shows the variation of the logarithmic creep strain rate with logarithmic stress for solid titanium alloy at various high temperatures during uniaxial creep. Considering uniaxial creep data obtained by Kim and Yang [21], creep constants of solid titanium alloy at various temperatures ranging from 500 to 950°C can be seen in Tab. I. .

\[
\dot{\varepsilon} = 10^3 \text{s}^{-1}
\]  

Fig. 4 shows the uniaxial stress–plastic strain response of titanium alloy at various high temperatures.
From Park [27], $f$ and $c$ in Eq. (14) can be written as, $f = \frac{1}{3}*(1-2\nu)$, $c = \frac{2}{3}*(1+\nu)$ respectively. From Eq. (14), the creep strain rate $\dot{\varepsilon}_y$ can be written as,

$$\dot{\varepsilon}_y = D\sigma_{eq}^{n-1}\left[(1+\nu)S_y + \frac{1-2\nu}{3} I_1 \delta_y\right]$$

(20)

Thus, obtaining the statistical relationship between Poisson’s ratio and the relative density of porous body is essential to construct such a constitutive model whose parameters
Values of Poisson’s ratio of the porous body $\nu$ can be calculated by axial strain and radial strain from uniaxial compression of titanium alloy powder compact using Gleeble1500 tester. The statistical relationship between Poisson’s ratio and relative density of porous body can be written as,

$$\nu = 0.5D^3$$

Moreover, the statistical relationship between Rockwell hardness and the relative density of porous body is essential to obtain relative density distributions of porous body. The statistical relationship between Rockwell hardness and relative density can be written as,

$$D = 5 \times 10^{-3} HRB + 0.45$$

### 4.4 Finite Element Simulation

To analyze the thermo-plastic densification behaviour of the titanium alloy powder under Hip, a hybrid model consisting of Eq. (6), Eq. (7), Eq. (8) and Eq. (9) were implemented into a finite element program (MSC.Marc), and the constants $q_1=1.25$ and $q_2=0.95$ were used in Eq. (8) as suggested by Becker et al. [28]. We also implemented the thermo-plastic response of solid titanium in Eq. (17) into the user subroutine of MSC.Marc. To analyze the creep densification behaviour of the titanium alloy powder under Hip, Eq. (14), Eq. (18), Eq. (19) and data in Tab.I were implemented into a finite element program (MSC.Marc). Fig.7 and Fig.8 show finite element meshes, applied pressure, and container dimensions for Hipping of titanium alloy powder. Because the model is axisymmetric body, one eighth model of the compact was used. The finite element calculations were obtained by assuming that a powder compact has a tap density of 0.65 (i.e. in Eq. (7), Eq. (18) and Eq. (19), $D_0=0.65$) and the friction coefficient $\mu=0.3$ between the powder and die walls during Hipping. The elastic response of titanium alloy at various high temperatures was obtained from the literature [21]. The relative density of the powder compact in the finite element analysis was obtained from the volume average of relative density at each element [23]. Thus,

$$D_{avg} = \frac{\sum_{j=1}^{m} D_j V_j}{\sum_{j=1}^{m} V_j}$$

Fig.9 and Fig.10 shows comparisons between the experimental data and the finite element calculations for relative density distributions of five points, which were selected from the revolution surface of workpiece Hipped under such a conditions that the temperature upped to 850°C and the pressure upped to 60MPa also at even rates within 40 minutes. The finite element calculation using the model of Fleck et al. and Gurson agrees well with the experimental data of the titanium alloy powder, however, using the model of Abouaf overestimates the experimental data.
Fig. 8. Dimensions of container used in Hipping

Fig. 9. FE calculation results using different densification models

Dimensions of Hipped container can be seen in Fig. 11 and Tab. II. shows comparisons between the experimental data and the finite element calculations for dimensions change of container. Values of container dimension, $L_1$, $L_2$, $K_2$, et al. are smaller using the model of Abouaf than Fleck et al. and Gurson, which agreed well with the finite element calculations for relative density distributions.
The main reason why the Abouaf model overestimated the experimental data is that the relationship between $f$, $c$ and relative density $D$ derived from plastic theory of only uniaxial cylinder compression is not accurate enough because of great difference in stress state between uniaxial cylinder compression and Hip. Besides, four possible explanations have been given to explain the differences between the experimental data and the finite element calculations not only using the Abouaf model but also the Fleck et al. and Gurson model: firstly, considering creep exponent $n$ relating to temperature (as shown in Tab. I.) is not enough, which also relates to equivalent plastic strain. Secondly, a microstructure source affects the constitutive behaviour of the powder. Thirdly, the compression tests were conducted on hipped compacts, but the powder was loose/unsintered for Hip. Last, the nature of the interparticle contacts and relative motion of particles during consolidation also affect the constitutive behaviour of the powder.

![Fig. 11. Container dimensions after HIP](image)

**Tab. II.** Container dimensions measured under different conditions

<table>
<thead>
<tr>
<th></th>
<th>$L_1$/mm</th>
<th>$L_2$/mm</th>
<th>$L_3$/mm</th>
<th>$L_4$/mm</th>
<th>$K_1$/mm</th>
<th>$K_2$/mm</th>
<th>$K_3$/mm</th>
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</thead>
<tbody>
<tr>
<td>before HIP</td>
<td>323</td>
<td>323</td>
<td>110</td>
<td>110</td>
<td>90</td>
<td>90</td>
<td>90</td>
</tr>
<tr>
<td>after HIP</td>
<td>303.69</td>
<td>293.05</td>
<td>102.46</td>
<td>109.29</td>
<td>78.53</td>
<td>78.98</td>
<td>82.09</td>
</tr>
<tr>
<td>Fleck et al. and Gurson</td>
<td>303.14</td>
<td>293.42</td>
<td>97.38</td>
<td>105.21</td>
<td>79.42</td>
<td>79.14</td>
<td>82.42</td>
</tr>
<tr>
<td>Abouaf model</td>
<td>302.05</td>
<td>291.96</td>
<td>97.04</td>
<td>106.76</td>
<td>76.45</td>
<td>81.33</td>
<td>81.30</td>
</tr>
</tbody>
</table>
5. Conclusion

(1) The present paper reports on the densification modeling of titanium alloy powder during Hipping. Experimental data were compared with the finite element calculations by using the model of Abouaf and the hybrid model consisting of Fleck et al. and Gurson, respectively. The finite element calculations by using hybrid model agreed well with the experimental data for densification behaviour of the titanium alloy powder under Hip. The finite element results by using the model of Abouaf, however, overestimated the experimental data.

(2) In order to easily obtain relative density distributions of porous body, the statistical relationship between Rockwell hardness and relative density has been formulated.

(3) The statistical relationships between the parameters \((f, c)\) and relative density \(D\) of porous body are given to such a constitutive model that equivalent plastic stress (or flow stress) of porous body relates to the first stress invariant and the second deviatoric stress invariant.

(4) The statistical relationships between Poisson’s ratio and relative density of porous body are given to such a constitutive model that relates to Poisson’s ratio of porous body.

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Reference