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An optimization approach for hot compaction technology of Mg–10Gd–2Y–0.5Zr alloy during solid-state recycling

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ABSTRACT

Solid-state recycling by hot extrusion is a new recycling method for machined chips. There are many factors which contribute to the complexity of hot compaction technology of chips. It is very important to obtain relative high-density blocks from chips and optimize these process parameters during the solid-state recycling. However, the process parameters are interdependent, and optimization of the combination of processes is time-consuming. In this work, the nonlinear relation of the temperature, the press and deformation velocity was established according to the rheology of the matrix material using a thermal simulation machine and mathematic regression analysis. Based on the experimental results of densities of blocks from chips, the hot-compacted model was built. The lowest energy consumption as criterion was also introduced to further optimize hot compaction parameters in both direct and indirect solid-state recycling means. The approach was found not only to obtain high-density blocks from chips, but make estimate on energy consumption during the hot-compacted stage. Especially, when the work velocity of the hydrostatic machine has severe influence on rheology of powders and phase transformation doesn't happen in high temperature environment, the method can quickly help engineers make the optimum hot-compacted technology of the powders.

1. Introduction

Recently, solid-state recycling by hot extrusion has been proposed as a new recycling method for machined chips because its cost is relatively low, also favorable for environment protection [1]. This solid-state recycling was divided into two forming ways according to characters of hot-compacted stage. One was a direct [1–5], and the other was an indirect [6–12] forming way. Just as powder metallurgy technology, this solid-state recycling for chips includes comminuting of chips, cold pre-compaction, pressureless or pressure sintering, and subsequently hot deformation such as sinter forging or hot-pressing [13–16]. The aim of hot-compacted processing is to obtain high-density blocks from chips. High ratio pore in blocks can't be entirely removed by hot deformation. Whether or not to obtain high-density blocks from chips is important for the solid recycling.

Y. Chino et al. [1–5] suggested that chips were directly extruded with high extrusion ratio after they were heated to the given temperature and kept for some minutes in the direct forming way. The pressure during extrusion was the same to the work pressure of the hydrostatic machine during hot-compacted stage. Densities of blocks from chips had a saturation value with the increasing of the press, which meant some energy was wasted. In the indirect forming way [6–12], M. Mabuchi. et al. suggested that a sintered compact was processed from machined chips at sintering temperature of 643 K with a pressure of 100 MPa, which didn't cover the effect of work velocity of the hydrostatic machine upon the rheology of chips in high temperature environment. J.Y. Wang. et al. suggested the fresh metal chips were pressed using a press machine of 100 ton capacity at room temperature, and the green relative density of the workpart after pressing was 88% of the fully condensed solid. In high temperature environment, the effect of work press of the hydrostatic machine on densities of blocks from chips wasn't considered.

Through the reviews of the literatures, it clearly reveals that there are many factors such as the temperature, the work press and velocity of the hydrostatic machine which contribute to the complexity of the hot compaction technology during solid-state recycling. However, these factors are interdependent, and optimization of the combination of processes is time-consuming. So far, the hot-compacted technology of chips is obtained by experience accumulation or massive experiments. It is necessary to find an approach not only to obtain high-density blocks from chips, but make estimate on energy consumption during the hot-compacted stage.

The theoretical models of densification proposed by some researchers in earlier studies on powders [17–19], however, have a limitation of only being applicable to the hot compaction processing of

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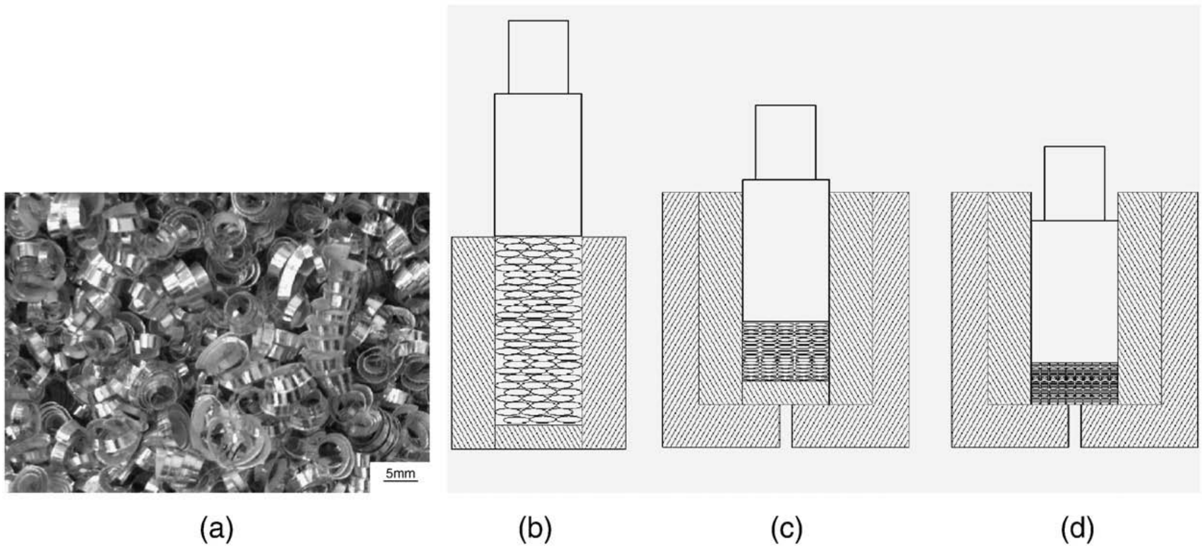

Fig. 1. Diagram of an indirect solid-state recycling facility and procedure: (a) loose chips, (b) cold compaction, (c) hot compaction and (d) hot deformation.

chips. The researchers were mainly interested in the density that would be obtained with a given temperature and a given pressure and in the stress and density distribution in a given mold. For the machined chips with the average geometric dimensions of ~12 mm × 4 mm × 80 μm, which is different from other powders, the effect of the work velocity of the hydrostatic machine on the densities of blocks needs considering during the hot-compacted stage.

Shima and Oyane proposed a plastic yield function for a porous solid by determining the parameters in their yield function empirically from uniaxial compression test of sintered porous copper and

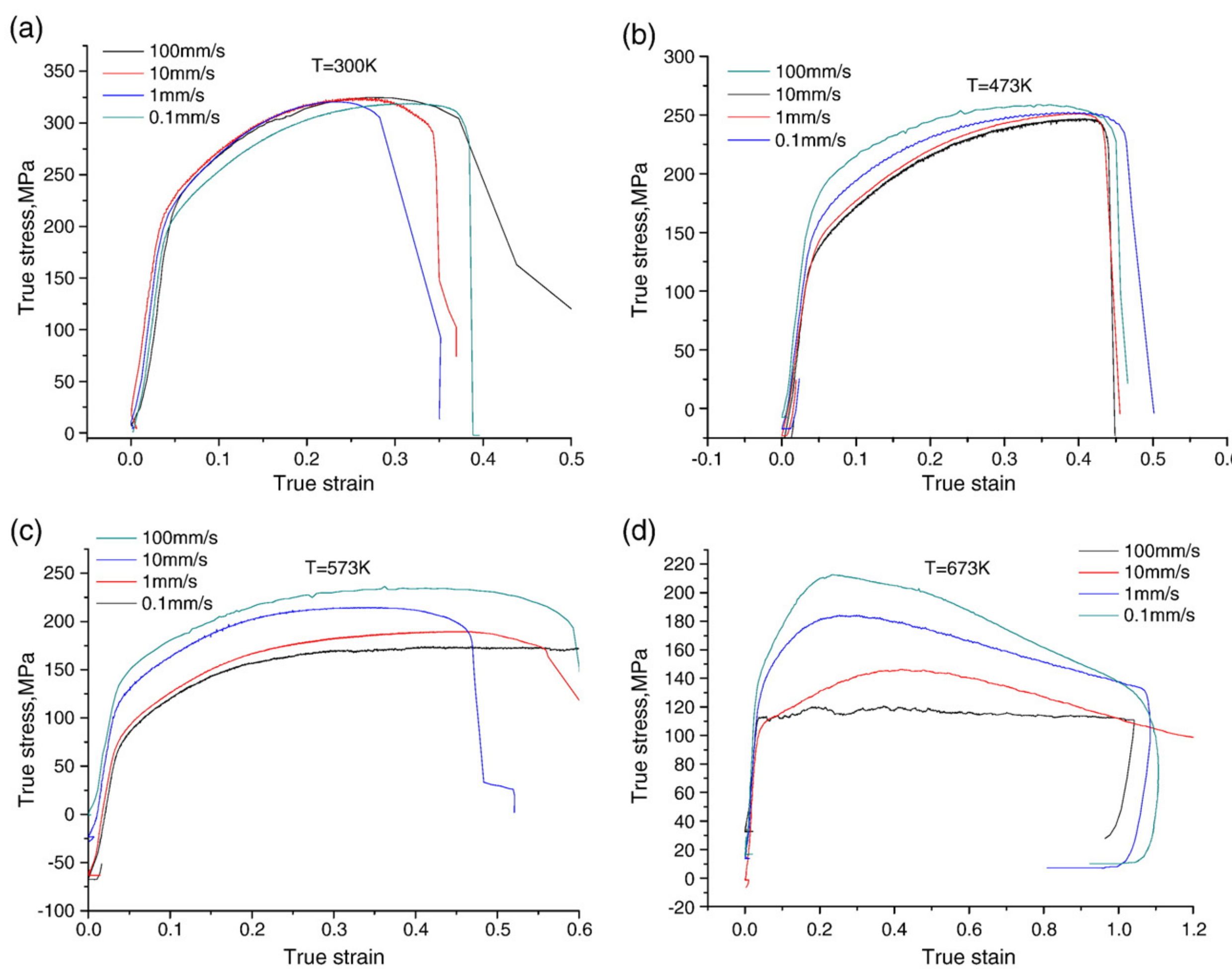

Fig. 2. The true stress–strain curves of the original GW102K alloy with various ram speed at (a) 300 K (b) 473 K (c) 573 K (d) 673 K, respectively.

iron [20]. The relative density of powder during the cold compaction was related to the compressive stress and the elastic modulus tensor of the matrix material. Lee and Kim proposed the Cap model by employing the parameters involved in the yield function of sintered metal powder and volumetric strain evolution under cold isostatic pressing [13]. C.S. Kang. et al. developed this model [21]. Densification behavior of iron powder by the developed model showed that the relative density of powder during the cold compaction was not only related to the compressive stress, but to the effective plastic strain rate of the matrix material. But this model wasn't involved in effect of temperature on relative density of powder compact at high temperature because of phase transformation of nanocrystalline titanium powder compacts.

In this work, phase transformation during hot compaction from chip didn't happen at high temperature. According to densification analysis on iron powder during cold compaction based on the rheology of the matrix materials [13,20,21], the nonlinear relation of the temperature, the press and deformation velocity have been established using a thermal simulation machine and MRA. Based on the experimental results of the densities of blocks from chips, the hot-compacted model was built. The lowest energy consumption as criterion was also introduced to further optimize hot compaction technology in both direct and indirect solid-state recycling means. This approach was found not only to obtain stable high-density blocks from chips, but make estimate on energy consumption during the hot-compacted stage.

2. Experimental procedures

2.1. Direct and indirect forming means

Chips with the average dimensions of ~12 mm × 4 mm × 80 µm were prepared by machining a Mg–10Gd–2Y–0.5Zr (Mg–9.95 wt.% Gd–2.3 wt.%Y–0.46 wt.%Zr) alloy cast ingot in a lathe. Fig. 1a showed machined chips of the alloy. Diagram of an indirect solid-state recycling facility and its procedure are shown in Fig. 1b. Firstly, the chips was filled into a cylindrical container with the dimension of ϕ30 and converted into a low-density block using a press of 200 MPa at room temperature. The green density of the block was 1.34 g/cm^3 by cold-compaction, which was 70% of the fully condensed solid of 1.91 g/cm^3. Secondly, these chips held for some minutes at high temperature were further converted in high-density blocks by hot compaction. Finally, these high-density blocks were deformed by hot processing after a gasket was removed.

In a direct way, where the gasket wasn't used, chips were directly hot-compacted after they were heated to the given temperature and kept for some minutes. The sintered blocks were extruded with a high ratio such as 45:1 [5] and 1600:1 [1]. In this study, without regard to chips missing during the hot-compacted stage, the processing from initial loose chips to high-density blocks at the pressure of the hydrostatic machine was considered. In the present work, the indirect forming means was use to check the hot compaction parameters during solid-state recycling.

2.2. Thermal simulation experiment and measurement of density

In order to establish the nonlinear relation of the temperature, the press and deformation velocity, the original ingot was used to reveal the compression rheology of chips. In this paper, a Gleeble 3500 thermal simulation machine was used to determine the flowing stress curves of the original ingots with the different temperatures and deformation velocities. Cylindrical ingots with the dimensions of ϕ10 mm × 15 mm were prepared for compression tests. In this simulation, a constant ram speed of 100 mm/s, 10 mm/s, 1 mm/s and 0.1 mm/s, respectively, was imposed according to characters of the work velocity of a hydrostatic machine. Fig. 2 showed the stress–strain curves of the original ingots under compression at 300 K, 473 K, 573 K and 673 K, respectively.

The densities of the hot-compacted blocks from chips were determined by the Archimedes' method. The relative density of a block was obtained based on the value of its density and the original ingot's.

3. Results

3.1. Rheology of the original materials

3.1.1. Multiple regression analysis (MRA)

Fig. 3 showed simple regression analysis results between the natural logarithm of the maximum compressive strengths (ln σ) vs. of ram speed (ln V) at 300 K, 473 K, 573 K and 673 K, respectively. Correlation coefficients and linear equations of ln σ vs. ln V with various deformation temperatures were also shown in Table 1. Values of the correlation coefficients were very near to 1, which represented a perfect correlation between ln σ vs. ln V. Besides, the mean square error $\left(\frac{1}{m}\sum\delta^2\right)$ between the experimental and predicated value of ln σ were little, which indicated that the established equations had great accuracy in predicting the maximum compressive strengths of the original ingot with the different ram speed.

Multiple regression analysis (MRA) is one of the most widely used statistical techniques for analyzing multifactor data [22]. According to characters of the equations of ln σ vs. ln V at constant temperature,

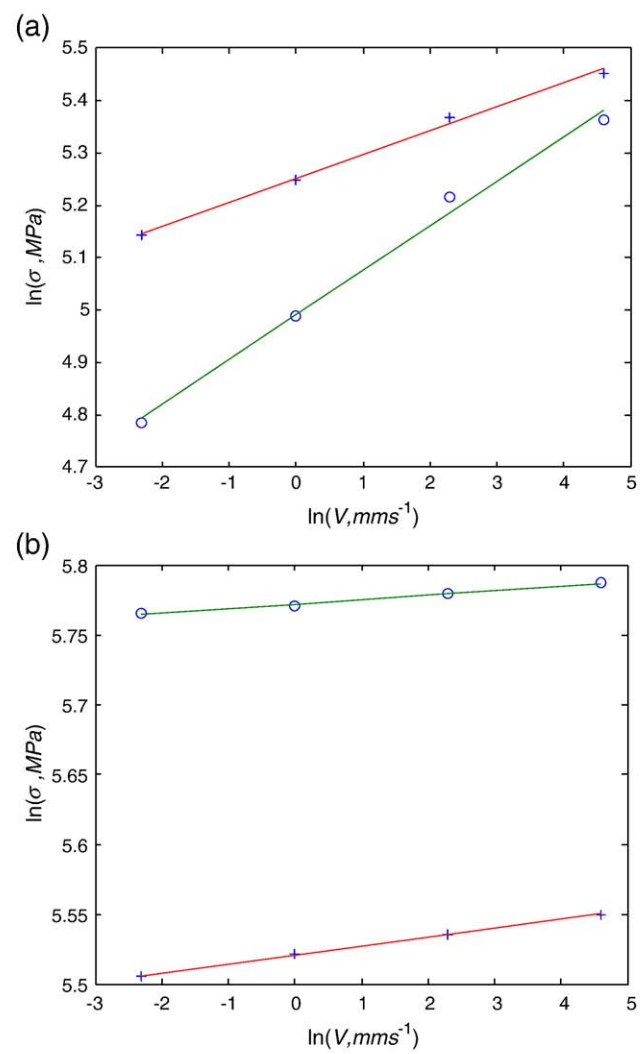

Fig. 3. Linear relations of ln σ vs. ln V at (a) 300 K, 473 K and (b) 573 K, 673 K, respectively.

Table 1
Linear equations and coefficient of correlation of ln σ vs. ln V, and error.

T/K	Equation modeling correlation between ln σ and ln V	Coefficient of correlation (r)	Mean square error $\left(\frac{1}{m}\sum\delta^2\right)$
300 K	$\ln\sigma_1 = 5.7718 + 0.0032\ln v$	0.958	6.45×10^{-7}
473 K	$\ln\sigma_2 = 5.5206 + 0.0064\ln v$	0.946	4.52×10^{-7}
573 K	$\ln\sigma_3 = 5.2510 + 0.0455\ln v$	0.934	7.3×10^{-2}
673 K	$\ln\sigma_4 = 4.9904 + 0.0851\ln v$	0.932	3.25×10^{-4}

the general expression for standard, linear MRA with various deformation temperatures can be written as

$$\ln\sigma = A(T) + B(T)\ln v \tag{1}$$

where σ is the dependent variable, T, V are two independent variables, $A(T)$ and $B(T)$ are the temperature-dependent coefficients.

Simple regression analysis between the coefficient $A(T)$ and coefficient $B(T)$ vs. T were plotted in Fig. 4, respectively. Mathematical expressions of $A(T)$ and $B(T)$ vs. T can be written as

$$A(T) = -0.0021\times T + 6.4411 \tag{2}$$

$$B(T) = 1.482\times10^{-4}\exp(0.0094T) \tag{3}$$

where mean square error $\left(\frac{1}{m}\sum\delta^2\right)$ between the experimental and predicated values according to Eqs. (2) and (3) is only 2.1×10^{-3}, 5.428×10^{-6}, respectively.

3.1.2. Confirmation test

Fig. 5 showed the verifying results for the predicated press using the established model for the original ingots. Mean square error $\left(\frac{1}{m}\sum\delta^2\right)$ and the maximum error $(\max|\delta_k|)$ between the experimental and predicated value of ln σ was 2.4×10^{-3}, 0.0866, respectively. The verifying results showed that the established model took on optimal generalization performance, and had great accuracy in predicating the maximum compressive strengths of the ingot with the different temperatures and deformation velocities.

3.2. Establishment of the model and experimental test

According to the predicated σ with the different work velocity of the hydrostatic machine (V) and hot-compacted temperature (T), the chips was compacted. Fig. 6 showed the relative densities of blocks from chips. Although the densities of blocks weren't very high, it is interesting to notice that their values were stable with the different V and T. For the relative density of 88% using the maximum compressive strength, it is indicated that this approach is reasonable, when the work press of the hydrostatic machine is modified, to get high-density blocks from chips. In a result, the model for hot compaction technology can be written as

$$p = a\cdot\exp\left[1.482\times10^{-4}(\ln V)\exp(0.0094T) - 0.0021T + 6.4411\right] \tag{4}$$

where p is work press of the hydrostatic machine in MPa, α is the modified coefficient, T is the heating-up temperature in K, T_0 is ambient temperature in K, ln V is the natural logarithm of work velocity (V) of the hydrostatic machine.

When α is 1.2 (i.e. the press was modified 1.2 times higher than the maximum compressive strength of the original ingots with the same V and T), the relative densities were increased to about 93% from 88% as shown in Fig. 6, which validated that it was credible to obtain stable high-density blocks from chips according to the model.

For magnesium alloys, the effect of work hardening on the compressive strength of the material is obvious in high temperature environment. In general, the higher work velocity of the hydrostatic machine, the higher hardness of the material, and thus the press is correspondingly increased.

3.3. Optimization of the model

3.3.1. In the direct solid-state recycling

The lowest energy consumption as criteria was introduced to optimize hot compaction technology in both solid-state recycling means. Energy consumption during the hot-compacted stage includes mechanical and endothermic power. Mechanical power is consumed when chips are inverted from the loose to compacted state by the hydrostatic machine. Endothermic power is also consumed when chips are heated. Assuming that the pressure in these experiments is constant during the compaction stage, total energy consumption, mechanical and endothermic power in this stage can be written as

$$w_{\text{direct}} = w_1 + w_2 \tag{5}$$

$$w_1 = ps(h_0 - h_1) \tag{6}$$

$$w_2 = mc\Delta t = mc(T - T_0) \tag{7}$$

where w_{direct} is total energy consumption in kJ, w_1 is mechanical power in kJ, w_2 is endothermic power in kJ, p is work press of the

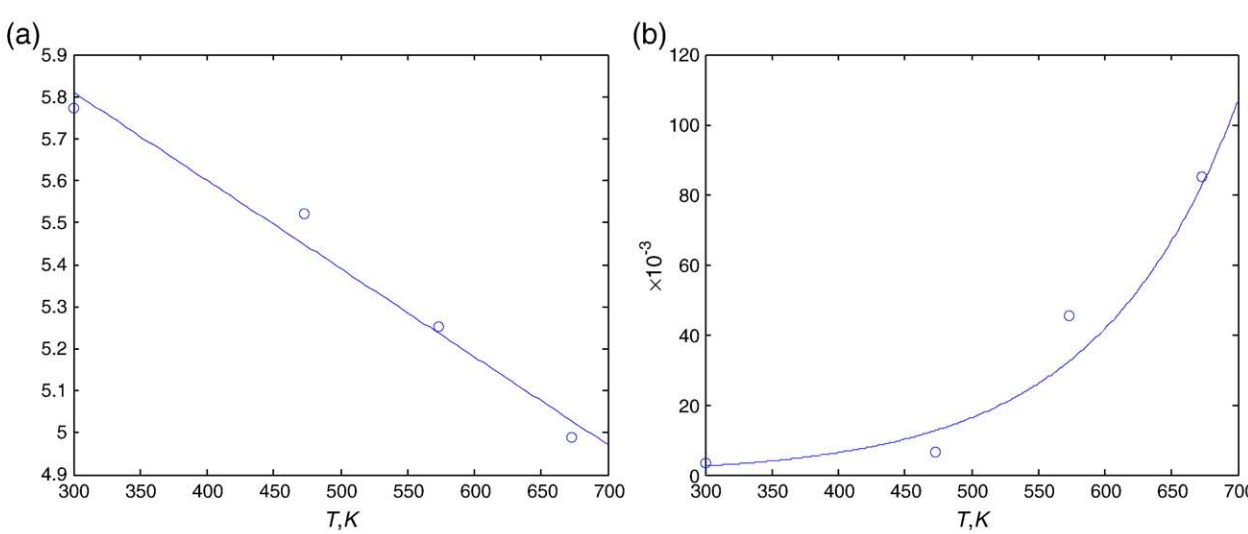

Fig. 4. Coefficient A (a) and coefficient B (b) vs. T.

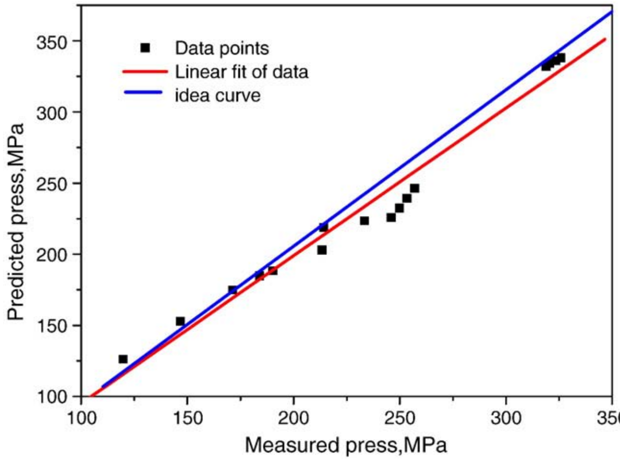

Fig. 5. Verifying results for the predicated press using the established equations by MRA.

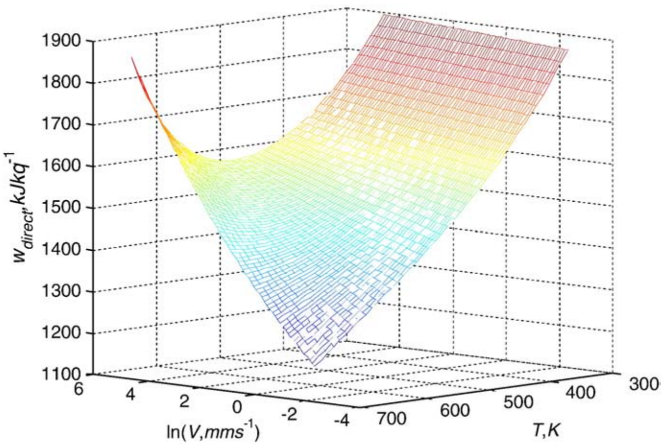

Fig. 7. Prediction on relationship of T and V to w_{direct} in a direct solid-state way.

hydrostatic machine in MPa, s is work area of press in m^2, h_1 is height of the hot-compacted block from chips in m, h_0 is height of chips in the loose state in m, m is mass of chips, c is specific heat capacity in kJ/kg·K, T is the heating-up temperature in K, T_0 is ambient temperature in K.

Assuming that the relative density of chips in the loose state is 10% in this experiment, and the relative density of block is 93% by hot compaction according to the model as above. For 1 kg of chips,

$$0.10\rho s h_0 = 1 \tag{8}$$

$$0.93\rho s h_1 = 1 \tag{9}$$

where ρ is the absolute density in kg/m^3, when value of h_0, h_1 is put in Eqs. (5)–(7), w_{direct} can be written as

$$w_{\mathrm{direct}} = 8.9247\frac{p}{\rho} + c(T - T_0) \tag{10}$$

For the Mg–10Gd–2Y–0.5Zr alloy, $\rho = 1.91\times10^3$ kg/m^3 and $c = 1.38$ KJ/(kg·K), $T_0 = 300$ K. According to the model in Eq. (4), when high-density (93%) blocks are obtained, total energy consumption during hot-compacted stage (w_{direct}) can be re-written as

$$w_{\mathrm{direct}} = 5.6071\exp\left[1.482\times10^{-4}(\ln V)\exp(0.0094T) - 0.0021T + 6.4411\right] + 1.38(T - 300) \tag{11}$$

where w_{direct} is total energy consumption in kJ, T is the heating-up temperature in K, V is work velocity of the hydrostatic machine in mm/s.

The prediction results in Figs. 7 and 8 according to the model show that:

(1) As V is 0.1 mm/s and T is 673 K, the lowest energy (1180 kJ/kg) for the GW102K magnesium alloy during the hot compaction stage is consumed (Fig. 7).
(2) w_{direct} varies complicatedly as T increases at a constant V. As V is 10–100 mm/s, w_{direct} decreases with T increasing to 520–620 K, and then increases as T further increases to 700 K. At V is 0.1–1 mm/s, w_{direct} decreases with T increasing. The lower V, the more quickly w_{direct} decreases (Fig. 8a).
(3) w_{direct} increases as V increases at any given T. The higher T, the more quickly w_{direct} increases. As T is 300 K, w_{direct} doesn't increase obviously (Fig. 8b).

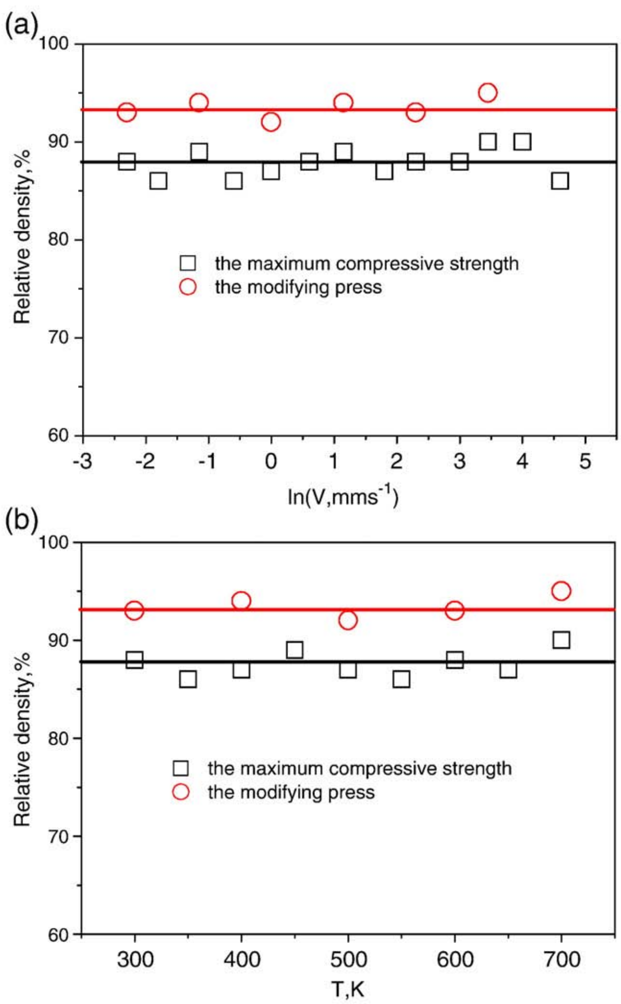

Fig. 6. Relationship between the relative density of blocks from chips and (a) V at 673 K and (b) T at a constant ram speed of 10 mm/s according to the maximum compressive strength of the original ingot and the modifying press.

3.3.2. In the indirect solid-state recycling

Total energy consumption, mechanical and endothermic power consumption can be written as

$$w_{\mathrm{indirect}} = w_1' + w_2' + w_3' \tag{12}$$

$$w_1' = p_0 s(h_0 - h_1') \tag{13}$$

$$w_2' = ps(h_1 - h_1') \tag{14}$$

$$w_3' = mc\Delta t = mc(T - T_0) = mc(T - 300) \tag{15}$$

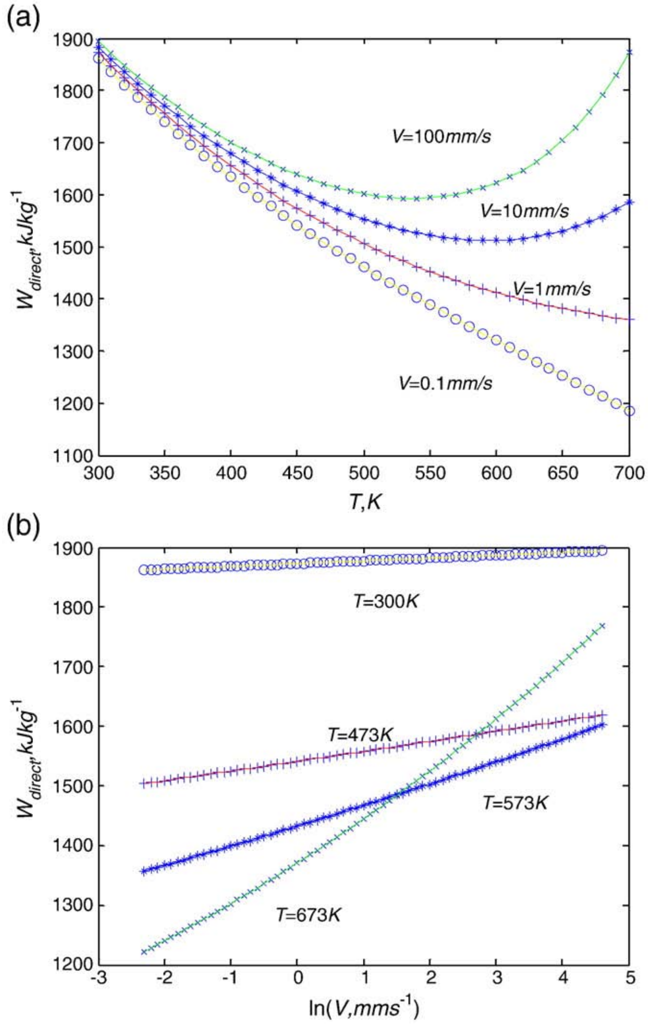

Fig. 8. Prediction for the effect of (a) V and (b) T on w_{direct} in a direct solid-state way.

where w_{direct} is total energy consumption in kJ, w_1', w_2' is mechanical power during the cold and hot-compacted stage in kJ, w_3' is endothermic power in kJ, p_0, p is work press of the hydrostatic machine during the cold and hot-compacted stage in MPa, s is work area of press in m^2, h_1 is height of hot-compacted block from chips in m, h_0 and h_1' is height of chips in the loose state and cold compaction

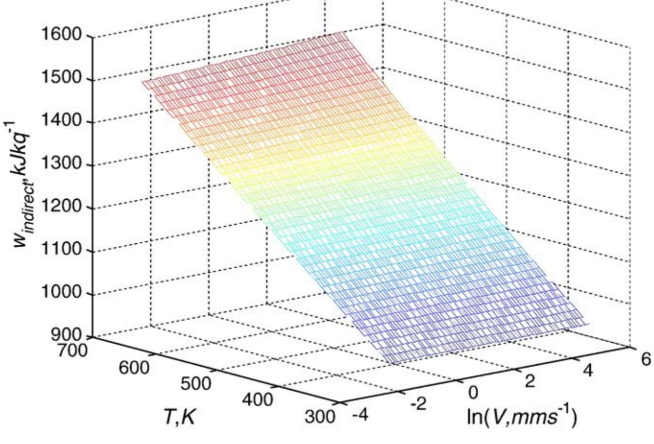

Fig. 9. Prediction on relationship of T and V to w_{indirect} in an indirect solid-state way.

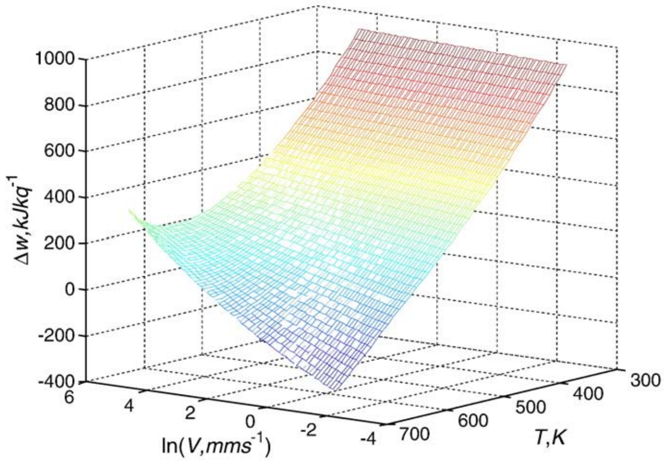

Fig. 10. Prediction on relationship of T and V to Δw.

in m, m is mass of chips, c is specific heat capacity in kJ/kg·K, T is the heating-up temperature in K, T_0 is ambient temperature in K.

The relative density of the cold-compacted block was 70% by cold compaction at the pressure of $p_0 = 200$ MPa in this experiment. According to the model in Eq. (4), where high-density (93%) blocks was obtained, w_{indirect} in the indirect way can be written as

$$w_{\text{indirect}} = 0.222\exp\left[1.482\times10^{-4}(\ln V)\exp(0.0094T) - 0.0021T + 6.4411\right] + 1.38T + 483.5288 \tag{16}$$

According to the model as above, the prediction results in Fig. 9 show that effect of work velocity of the hydrostatic machine on total energy consumption is negligible. During hot compaction, the higher temperature, the more total energy consumption. The lowest total energy (980 kJ/kg) for the GW102K magnesium alloy during the compaction stage is consumed as compaction temperature is 300 K.

3.3.3. Comparison between the direct and indirect solid-state recycling means

Δw is the difference between w_{direct} and w_{indirect}, whose expression can be written as

$$\begin{aligned}\Delta w &= w_{\text{direct}} - w_{\text{indirect}} \\ &= 5.3851\exp\left[1.482\times10^{-4}(\ln V)\cdot\exp(0.0094T) - 0.0021T + 6.4411\right] \\ &\quad - 897.5288\end{aligned} \tag{17}$$

The prediction results in Figs. 10 and 11 show that:

As V is 0.1–4.5 mm/s and T is 580–700 K, $\Delta w < 0$. More energy in the indirect way is consumed than in the indirect way during the hot

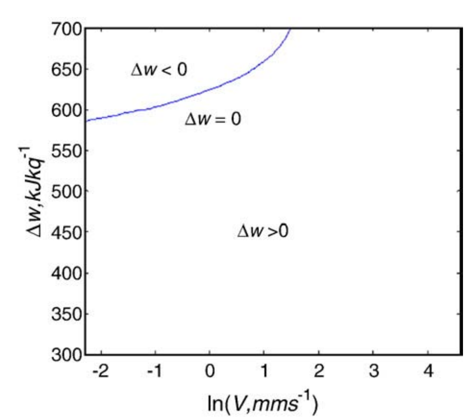

Fig. 11. Two-dimensional cross-section from $\Delta w = 0$.

compaction stage. A direct recycling way for chips should be elected according to the lower energy consumption.

4. Discussion

Up to now, the hot-compacted technology of chips is obtained by experience accumulation or massive experiments. There are many process parameters such as hot-compacted temperature, the work velocity and press of a hydrostatic machine which ascribed to the complicated rheology of machined chips during the hot-compacted stage. These process parameters are interdependent, and the work velocity of the hydrostatic machine is not taken attention to by some researchers. Besides, they suggested the different hot-compacted technology for the same alloy chips. No criteria were also defined for the optimum hot-compacted technology of chips. Therefore, it is necessary to find an approach to optimize hot compaction parameters of chips during solid-state recycling.

In this work, the nonlinear relation between the hot-compacted temperature and the work press and velocity of the hydrostatic machine was established according to the rheology of the matrix materials. Based on the highly stable relative density of blocks from the chips in experiments, the hot-compacted model was built. The lowest energy consumption as criteria was also introduced to optimize hot compaction technology in both direct and indirect solid-state recycling. The approach was found not only to obtain high-density blocks from chips, but make estimate on energy consumption during the hot-compacted stage.

According to the results as above, this optimization approach mainly included: establishment of rheology of the original materials, to choose an appropriate correction coefficient α according to the relative densities of blocks from chips, and the optimization of the model according to the lowest energy consumption. For this approach, it is noted that coefficient α is important to obtain high-density blocks from chips during the hot-compaction stage. Three factors influence on coefficient α are: (1) the friction between the chips and die wall, (2) relative density of blocks obtained from chips, and (3) the size of chips. Friction between a ram and dies could weaken the effect of pressure on the density of blocks from chips. The higher the relative densities of blocks are, the higher the coefficient α is. The coefficient α has an optimal value. When coefficient α is lower than this optimal value, the relative density would increase dramatically, up to increase slowly with increase of pressing. Correspondingly, this suggests that the optimal value of the coefficient α is near to a saturation value of blocks densities. Besides, to obtain the same density blocks from chips, the smaller the size of chips is, the smaller the press is, and correspondingly the smaller the coefficient α is.

5. Conclusions

It is very difficult to obtain theology of the chips. The nonlinear relation of the hot-compacted parameters such as the hot-compacted temperature, the work press and velocity of a hydrostatic machine has been established according to the rheology of the matrix materials using a thermal simulation machine and mathematic regression analysis. Based on relative densities of blocks in experiments, the hot-compacted model was built. The lowest energy consumption as criteria was also introduced to optimize hot compaction technology in both direct and indirect solid-state recycling. The approach was found not only to obtain high-density blocks from chips, but make estimate on energy consumption during the hot-compacted stage.

In the present study, the proposed approach can be applied for hot-compacted technology of various powders from nanometer to centimeter size. The smaller the size of chips is, the smaller the press is, and correspondingly the smaller the coefficient α is. Especially, when the work velocity of the hydrostatic machine has severe influence on rheology of powders and phase transformation doesn't happen in high temperature environment, the method can quickly help engineers make the optimum hot-compacted technology of the powders.

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