Chapter 2
Resistance Sintering

2.1 Principle and Physical Mechanisms of Resistance Sintering

Lenel [1] classified resistance sintering under pressure as a type of hot pressing. In resistance sintering under pressure, high currents pass through the powder compact at low applied voltages. In conventional hot pressing, the die is heated by radiation from resistive heaters, by high-frequency induction, or by passing an electric current through it. The material of the die needs, therefore, to be able to withstand temperatures required for high-temperature sintering and possess sufficient mechanical strength. In order to overcome these difficulties, one can heat only the material to be sintered not heating the die directly by passing a high current through the powder compact. In resistance sintering, the heat is generated within the powder and is not conducted from the die. Therefore, only conductive materials can be sintered by resistance sintering. Taylor suggested placing powders in a glass or ceramic tube between the plungers [2]. In a setup described by Lenel [1] (Fig. 2.1), a green compact or a loose powder is placed between wafers, which are made of a material that has a higher electrical resistivity, a lower thermal conductivity, and a higher melting point than the material of the plungers to make the heat distribution in the sample more uniform. If wafers are not used, then the heat is rapidly dissipated through the plungers, which are made of a highly conductive material, resulting in insufficient sintering of regions near the flat ends of the sample. The powder compact is isolated from the die by a ceramic liner.

Resistance sintering is performed when a direct current or an alternating current of low frequency passes through the powder compact. Pressure is applied when the compact is formed by loose powders; pressureless resistance sintering is also possible when electric current is applied to the compact pre-pressed or pre-sintered before the resistance sintering. The sintering time is usually short (a fraction of a second). The resistance sintered material is also cooled rapidly as the current is switched off. The principal requirement to the powder compact for its successful

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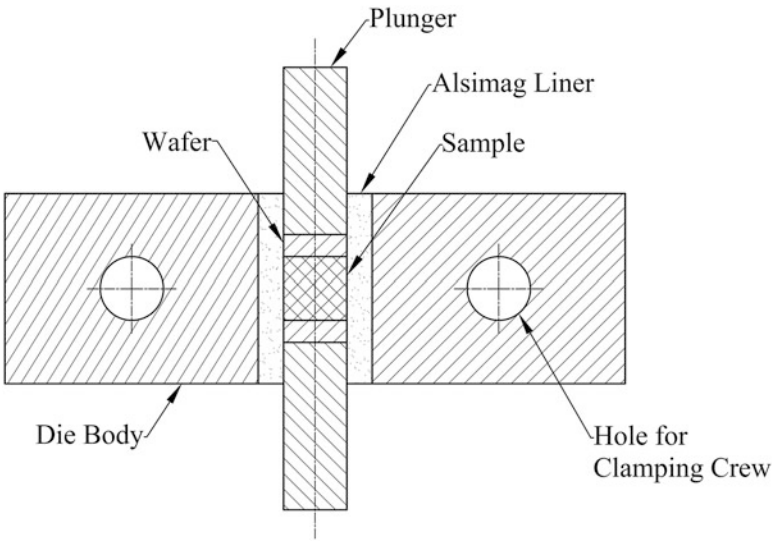

Fig. 2.1 Schematic of the die–plunger setup used for resistance sintering (Adapted from Lenel [1], Copyright (1955) with permission of Springer)

resistance sintering is a rather low resistance. The total resistance of the powder compact is determined by the resistivity of the material itself and contact resistances between the particles. The latter depends on the particle size distribution, the level of applied pressure, and the presence of oxide films on the surface of the particles. The electrical conductivity of the powder compact from carbonyl nickel powders increases with the applied pressure as p^n, where p is applied pressure and $n = 0.85$ [3]. In composite mixtures made of a conducting and an insulating component, the conducting component should form a continuous network within the compact. The sample with 0.1 Ω resistance will not be able to carry enough current for sintering to occur [1]. For materials with a high initial resistance, a two-pulse sintering process can be suggested for a better control of the changes in the sample's resistance during sintering and the microstructure development. Using a two-pulse technique, it is possible to benefit from different parameters of the pulses (time, voltage) chosen for different purposes. The first pulse is intended to break down the resistance, and the second pulse is real sintering. This technique allows avoiding overheating of the compact. Greenspan [4] described so-called impulse resistance sintering – the process designed to fully utilize the heat generated at the inter-particle contacts to bond the particles together. This is done by applying a very low pressure in the beginning of the process (6 MPa) and increasing it when the compact reached a plateau on the shrinkage curve. This method was quite successful in making tungsten compacts of 22% porosity, and despite that, possessing a high flexural strength, which indicated good bonding between the particles. It can be assumed, however, that the early formation of the well-established inter-particle contacts hindered densification of the compacts upon further sintering – this aspect was theoretically

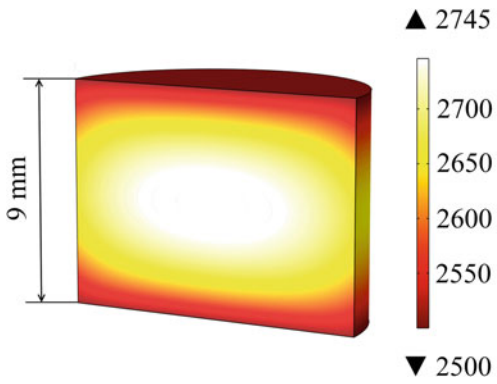

Fig. 2.2 Temperature distribution in an iron compact sintered by resistance sintering (Adapted from Lenel [1], Copyright (1955) with permission of Springer)

elaborated by Olevsky et al. in Ref. [5] – and was the reason for the difficulties of producing fully dense compacts by this technique.

The calculated temperatures resulting from the models can be verified experimentally by placing small pieces of metallic wire with different melting points in the compact [1]. The center of the compact has the maximum temperature (see example in Fig. 2.2 for an iron compact). Lower temperatures of the peripheral regions of the compacts can cause insufficient sintering. In order to achieve a better sintering uniformity, one of the wafers is made concave. A higher current passing through the edges where its path is shorter makes the overall temperature distribution more uniform.

For successful resistance sintering, the ratio of the length to diameter of the compact should not exceed 1. Larger ratios result in poor sintering of the central parts of the compacts. The grain size of the materials resistance sintered under pressure is usually smaller compared with conventionally sintered ones. Although the contact between the particles is established more rapidly during resistance sintering under pressure creating conditions for the grain growth to occur, the short sintering time prevents extensive grain growth. Due to the shortness of the sintering time, the degree of chemical homogenization is also limited in multicomponent mixtures during resistance sintering. The alloy formation can be arrested at a certain stage producing a metastable material from the phase composition and microstructure points of view. Lenel [1] states the shortness of the process of achieving high relative densities and the possibility of producing non-equilibrium microstructures as the main features and advantages of resistance sintering.

As electric current starts passing through the compact upon the application of voltage, the boundaries separating the material with increased electrical conductivity move from the contact surfaces of the compact and the punch (electrode) into the interior of the sample. The current increases dramatically when the two boundaries meet in the central part of the sample. The advantages of two-pulse sintering for compacts of high initial resistance are demonstrated in Fig. 2.3. When current is applied to a low initial resistance compact, it becomes constant after a few cycles

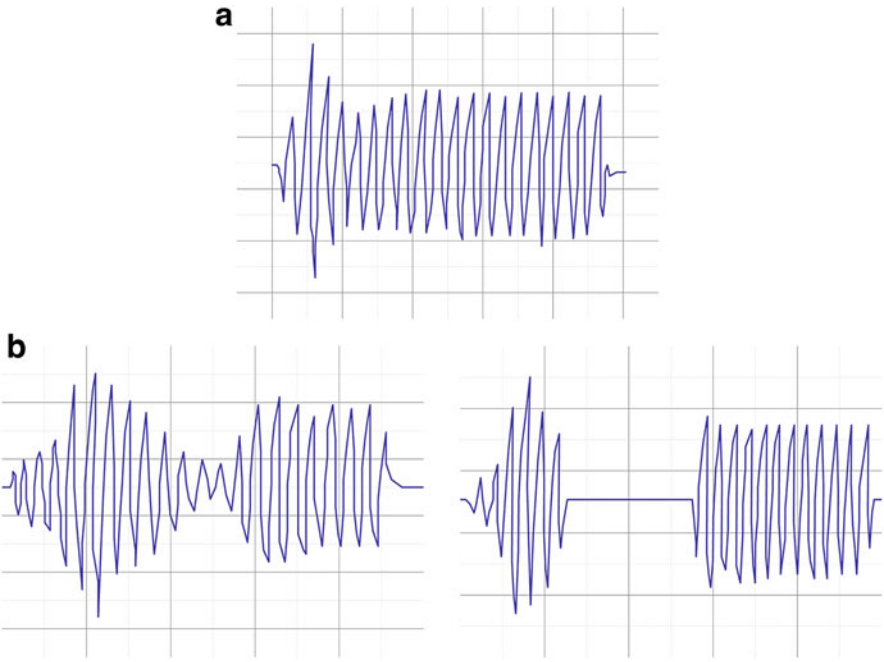

Fig. 2.3 Current traces for the case of a low initial resistance of the compact (**a**) and high initial resistance of the compact, the cases of one- and two-pulse sintering (**b**) (Adapted from Lenel [1], Copyright (1955) with permission of Springer)

because the current regulator can compensate the changes in the resistance. When a high-resistance compact is sintered by a single pulse, the resistance changes so dramatically that the current regulator is unable to follow these changes.

2.2 Resistance Sintering Equipment

Akechi and Hara [6] analyzed the evolution of contributions from the resistance of the powder and that of inter-particle contacts to the total resistance of a titanium compact sintered by resistance sintering using an alternating current of 50 Hz frequency. The analysis was performed based on the measured profiles of electric current and voltage (Fig. 2.4).

As is shown in Fig. 2.5, the following heat-generating elements can be designated: the inter-particle contacts in the powder compact, the powder particles themselves, the contact between the punches and the powder compact punches, the punches, the contact between the punch and the plunger, and internal elements of the setup. At the final stage of sintering, when bonding between the powder particles has been already established, the resistance of inter-particle contacts does not

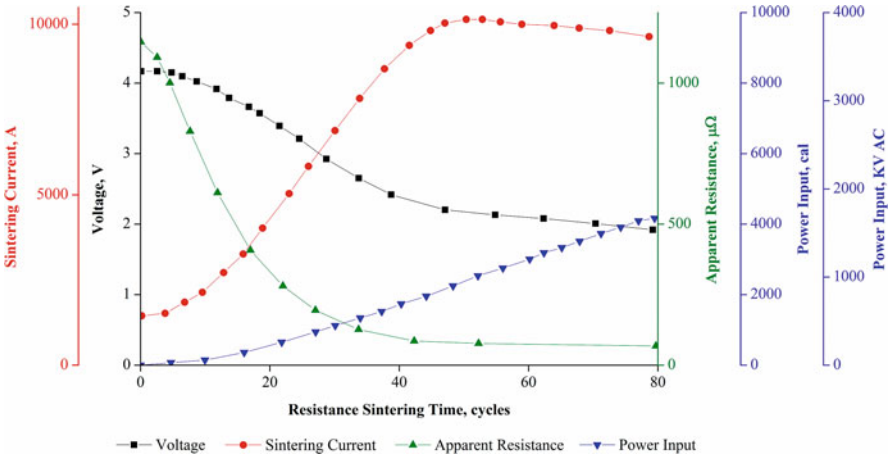

Fig. 2.4 Evolution of current, voltage, apparent electric resistance, and energy input with the sintering time for a resistance sintered titanium powder compact under pressure. Power input is given in calories and kV·A·cycle units (Reprinted from Akechi and Hara [6], Copyright (1977) with permission from Japan Society of Powder and Powder Metallurgy)

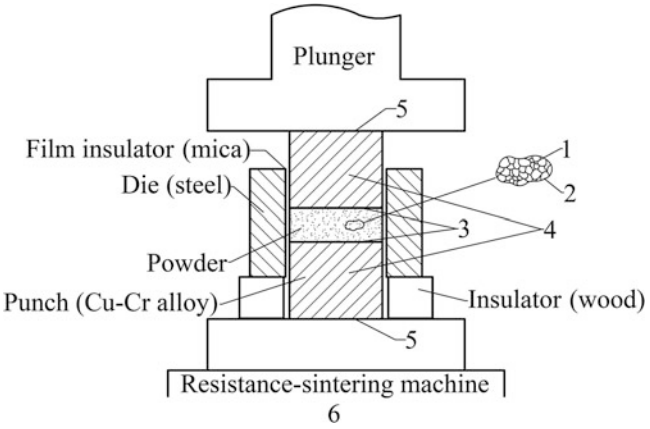

Fig. 2.5 Heat-generating elements of a resistance sintering setup: 1, inter-particle contacts; 2, powder particles; 3, resistance of the contact between the punches and the powder compact; 4, resistance of the punches; 5, resistance of the contact between the punch and the plunger; 6, internal resistance of the setup (Reprinted from Akechi and Hara [6], Copyright (1977) with permission from Japan Society of Powder and Powder Metallurgy)

contribute any longer to the total resistance of the setup. The resistance of the powder compact can be calculated using the resistivity of the bulk metal and dimensions of the compact. The temperature, to which the sample was heated at a certain moment of the sintering process, can be estimated from the measured net power and thus can be taken into account when calculating the resistance of the powder compact and the punches. The sum including the contact resistance between the punches and the

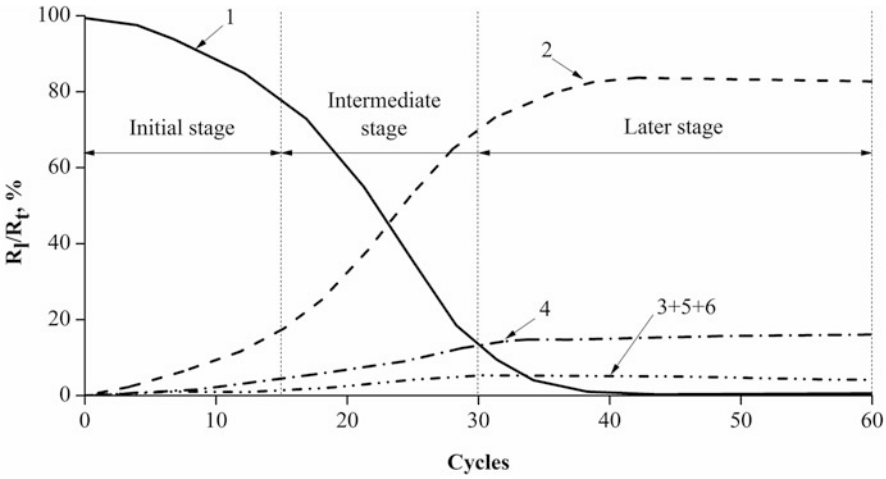

Fig. 2.6 Contributions of resistances of different heat-generating elements of the resistance sintering setup to the total resistance (notations are as in Fig. 2.5) (Reprinted from Akechi and Hara [6], Copyright (1977) with permission from Japan Society of Powder and Powder Metallurgy)

powder compact, the contact resistance between the punch, and the plunger and the resistance of internal elements of the setup can be calculated by subtracting the resistance of the powder compact from the total resistance at the final sintering stage. At the initial sintering stage, the compact is mainly heated due to heat evolution at the inter-particle contacts. At the intermediate stage, the heat is generated both within the particles and the contacts between them. In accordance with these considerations, Fig. 2.6 shows the evolution of the fractions of resistances of different heat-generating elements in the total resistance of the sintering setup.

2.3 Properties of Specimens Processed by Resistance Sintering

In experiments performed by Montes et al. [7], the resistance of a titanium powder compact decreased rapidly during 0.2 s from the beginning of the sintering process; this was accompanied by a certain decrease in the porosity of the compact. Upon further sintering, the resistance did not change, while the porosity continued to decrease (Fig. 2.7). Such behavior was explained by a fast resistivity reduction of oxide films of semiconductor nature with increasing temperature at the initial stage of sintering. Temperatures needed for significant material softening were not reached until the intermediate stage of sintering, during which the densification of the compact continued.

Akechi and Hara [6] emphasized the role of the positive coefficient of resistance in homogenizing the microstructure of the powder compact and stabilizing the

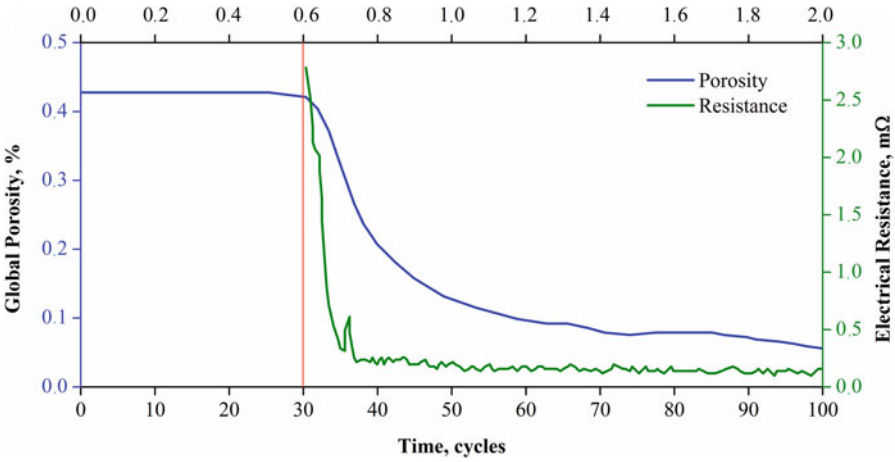

Fig. 2.7 Evolution of porosity and electrical resistance of the compact produced by resistance sintering of a titanium powder (passage of current started at the time equal to 30 cycles) (Reprinted from Montes et al. [7]. Copyright (2011) with permission of Springer)

process of resistance sintering. The regions of the powder compact of low resistance initially carry the most of the current; however, due to Joule heating, the resistivity of the material increases such that redistribution of current occurs. This redistribution causes better sintering of the previously poorly sintered regions of high initial resistance. Rykalin [8] suggests the "self-regulation" term for this phenomenon.

Being a fast sintering technique, resistance sintering does not require a protective atmosphere. However, when reactive metals, such as titanium, are resistance sintered, an increase in the oxygen and nitrogen content in the compact relative to that in the powder can be observed. Montes et al. [7] found that consolidation of a titanium powder with a mean particle size of 24 μm by resistance sintering (total duration of sintering was 1.4 s) results in an order of magnitude increase in the oxygen content, while the content of nitrogen doubles.

Since during the resistance sintering both the electric current and the sample's resistance change with time, in order to assess the contribution of the Joule heat in comparative studies of the microstructure and properties of the compacts sintered from the same powder under variable conditions of resistance sintering (current, number of cycles of current), the calculation of the thermal energy generated per unit mass of the powder (specific thermal energy) due to Joule effect is helpful:

$$\eta = \frac{1}{M} \int_0^t I^2(\tau) R(\tau) d\tau, \tag{2.1}$$

where $I(\tau)$ is the current, $R(\tau)$ is the electrical resistance, and M is the powder mass.

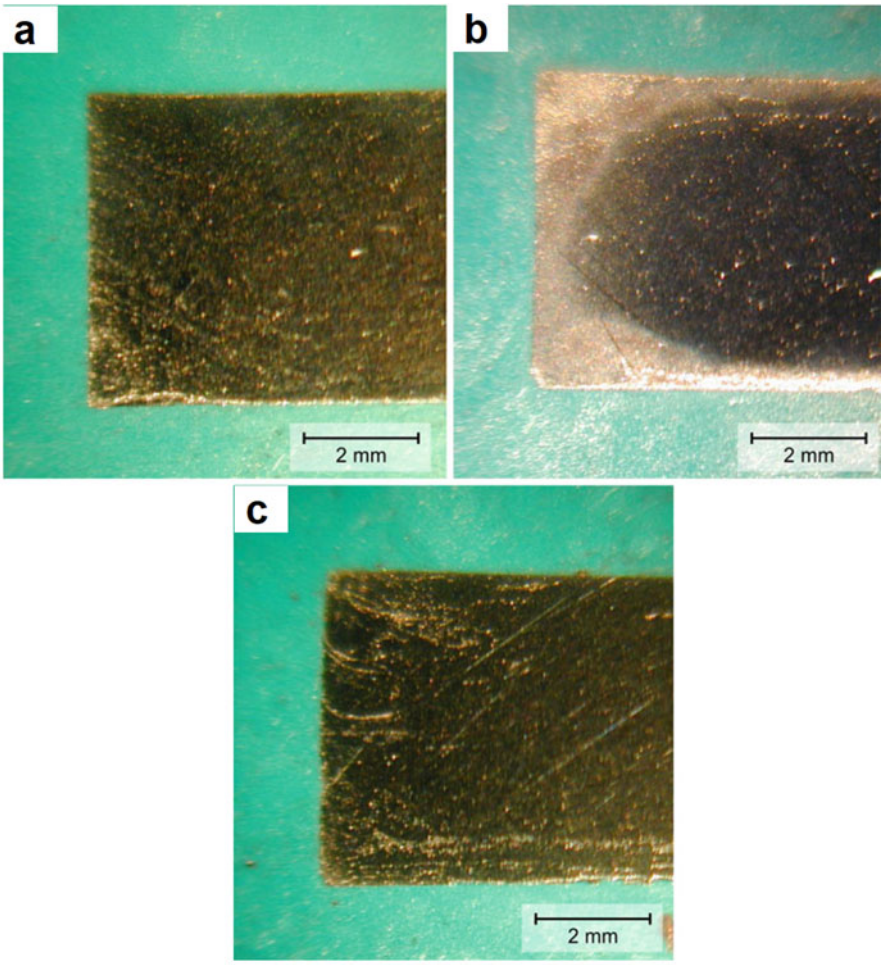

Fig. 2.8 Macrographs of diametrical sections of halves of compacts: (**a**) conventionally sintered (porosity 2%), (**b**) resistance sintered compact (4.0 kA, 50 cycles, porosity 15.8%), (**c**) resistance sintered (6.0 kA, 80 cycles, porosity 3.3%). A higher value of η for sample (**c**) resulted in a more uniform distribution of the porosity (Reprinted from Montes et al. [7]. Copyright (2011) with permission of Springer)

A higher value of η results in a more uniform distribution of the porosity in the resistance sintered compacts (Fig. 2.8). The highest temperature is reached in the center of the sample making it denser than the peripheral regions adjacent to the electrodes cooled during the process and walls of the die. These effects are more pronounced at lower η.

Montes et al. distinguish three types of pores in resistance sintered compacts. The first type is the porosity in poorly sintered regions; these pores are comparable in size with the initial powder particles (Fig. 2.9a). The second type of pores is the residual

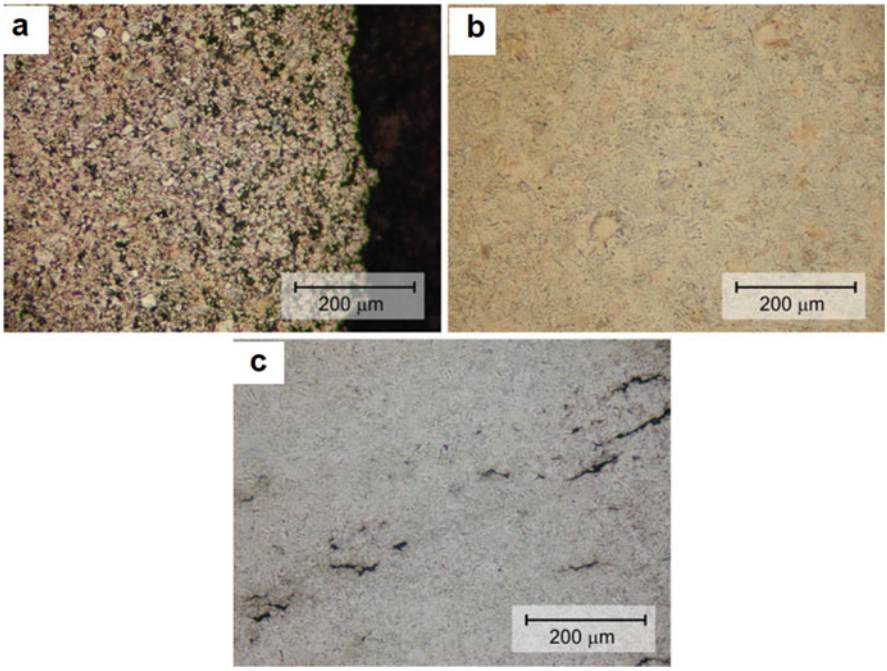

Fig. 2.9 Three types of pores in resistance sintered compacts: (**a**) pores in poorly sintered regions, (**b**) residual pores in well-sintered regions, (**c**) pores formed during pressing (shear cracks) (Reprinted from Montes et al. [7]. Copyright (2011) with permission of Springer)

pores in well-sintered regions (Fig. 2.9b). Pores of the third type are large pores that have the greater axis perpendicular to the pressing direction and can be considered as shear cracks introduced during pressing (Fig. 2.9c). These pores can be found only in samples sintered at high currents, as they form in a well-sintered material that does not allow any particle rearrangement in response to shear forces.

Thanks to short processing times and high cooling rates in resistance sintering, the resistance sintered compacts may show microstructures different from those of the conventionally sintered ones. In resistance sintered compacts heated above the temperature of the $\alpha \rightarrow \beta$ transition of titanium, lamellar grains of the α-phase form during fast cooling of the β-phase [7]. In conventionally sintered titanium during slow furnace cooling, large equiaxed grains of the secondary α-phase form.

According to Belyavin et al. [9], resistance sintering by DC current in the case of long process durations (longer than 1 h) can lead to the differences in the microstructure and grain size of the cathode and anode ends of the specimen and nonuniform distribution of porosity.

Lagos et al. [10] and Schubert et al. [11] presented a modified method of resistance sintering based on the action of a current of high density. In their processing, WC–Co powder was filled in a ceramic die between two copper electrodes. Sintering was performed in air with a holding time of only 500 ms

[10]. The current was produced by a low-voltage transformer (around 10 V). The sintered samples consisted of a dense core surrounded by a porous surface layer. The authors pointed out that, in contrast to spark plasma sintering, in which the processing time is in the range of minutes and a controlled atmosphere is needed, a fast single-pulse technique allows conducting sintering in air without oxidizing the material. This is a very important advantage, as it reduced the cost of the sintering equipment. Schubert et al. [11] compared the microstructure and properties of WC–Co materials consolidated by the fast resistance sintering and hot isostatic pressing and found that the former possessed finer microstructures and higher hardness.

2.4 Summary

In resistance sintering, the heat is generated within the powder and is not conducted from the die. Therefore, only conductive materials can be sintered by resistance sintering. During resistance sintering, temperature distribution within the sample is spatially nonuniform, as shown by experiments and modeling. During resistance sintering, the center of the compact has the maximum temperature, while peripheral regions of the compacts are prone to insufficient sintering. At the same time, the short sintering time in resistance sintering prevents extensive grain growth. In powder mixtures, it allows compositional homogenization to proceed only to a limited extent. A high potential of a recently suggested modification of resistance sintering – sintering by a current pulse of high density – should be noted. This variation of resistance sintering allows working in air and still avoiding oxidation of the material due to a very short duration of the pulse.

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