論 文

Microstructural Characteristics of Rapidly Solidified 304 Stainless Steel Powders Produced by Gas Atomization

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Abstract

가스분무장치를 이용하여 제조된 304 stainless steel 분말의 미세응고조직 특성을 투과전자현미경으로 관찰하였다. 분말이 sandwich 형상으로 존재하도록 구리로 전기도금한 후 tripod jig 를 이용하여 기계적 연마하여 TEM 시편을 제작하였다. 이 방법으로 제조된 TEM 시편은 넓은 지역에서 200 KV 로 가속된 전자가 투과하기에 충분히 얇았으며, 작은 분말의 경우에는 분말 전체를 관찰할 수 있었다. 제한시야회절법 (SADP) 을 이용하여 100 μm 이하 분말의 결정구조를 조사한 결과에 따르면 가스분무법으로 급냉응고된 대부분의 분말은 austenite 상으로 응고되었으며, 모든 austenite 분말은 크기에 관계없이 쌍정조직 (twin structure) 이 발견되었으며 그 밀도 역시 아주 높았다. 그러나 직경이 2 μm 이하의 분말에서는 큰 과냉 (supercooling) 효과에 의하여 준결정상인 bcc 상으로 응고됨을 발견하였다. (Received April 16, 2001)

Keywords : 가스분무법, 304 stainless steel 분말, tripod jig, 급냉응고, twin structure, supercooling

1. Introduction

Processing of materials by rapid solidification can impart significant benefits compared to conventional ingot metallurgy technique. Improved chemical homogeneity, microstructural refinement, extended solute solubility, and the retention of metastable phases can be realized[1]. Rapid solidification of 304 stainless steel is of particular interest because of its importance as a commercial alloy. Kelly et al. have studied mechanical properties of the rapid solidification processed 304 stainless steel and compared with those of a conventionally processed 304 stainless steel[2,3]. Significant improvements in tensile strength were observed after the rapidly solidified powders were consolidated at relatively high temperature. These observations suggest that microstructural characteristics formed in powders during the rapid solidification processing have been retained or retarded at the consolidation temperature. There is considerable interest in examining the characteristic features of the powders because they are directly responsible for the mechanical properties of the rapidly solidified alloy.

In this study the microstructure was investigated for rapidly solidified 304 stainless steel powders produced by inert gas atomization process. Most of examination performed in transmission electron microscope(TEM). Especially, mechanical polishing was used to prepare TEM specimens of the powders[4]. This method have advantages of being able to prepare large electron transparent area enough to examine the whole area of small powders and reduce the radiation damage introduced by ion milling.

2. Experimental Procedure

In the inert gas atomization process it is involved pressurizing and mechanical mixing of the melt with argon. The melt is blown into a low pressure chamber with argon. The molten droplets are produced by the carrier argon associated with the melt stream cools under vacuum or low pressure of argon. The powders of less than 100 μm diameter were sieved for the examination of solidification structures. There are two

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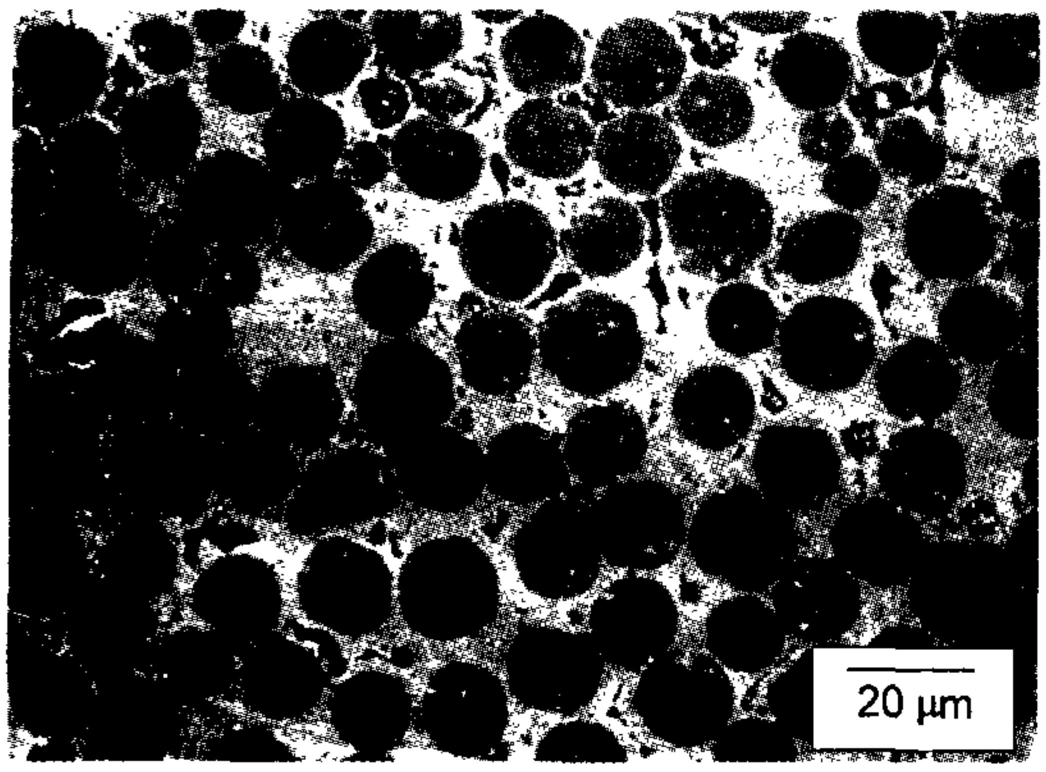

Fig. 1. SEM micrograph showing distribution of 304 stainless steel powders in electroplated copper.

steps in preparing TEM specimens of as-solidified 304 stainless steel powder particles without chemical etching or thermal treatment. Both of these treatments produce undesirable artifacts on the TEM specimen. First, it is necessary to make a foil of powder embedded in an electroplated metal. The foil is then thinned to electron transparency by mechanical polishing. A monolayer of powder particles on an electroplated copper foil is polished to ensure exposure of half of the sphere as shown in Fig. 1. The polished side is then glued to the tripod jig. The second side is polished on the diamond lapping films with the angle resulting from about a 100 to 200 μm advancement of the rear micrometers on the tripod jig. This creates a wedge shaped sample and the leading edge may be very thin. After polishing finally with syton to the thickness of electron transparency, a TEM support grid is mounted to the specimen. This specimen has been cleaned for about 10 minutes by ion milling. Transmission electron microscopy was performed using a JEOL 200CX-II microscope.

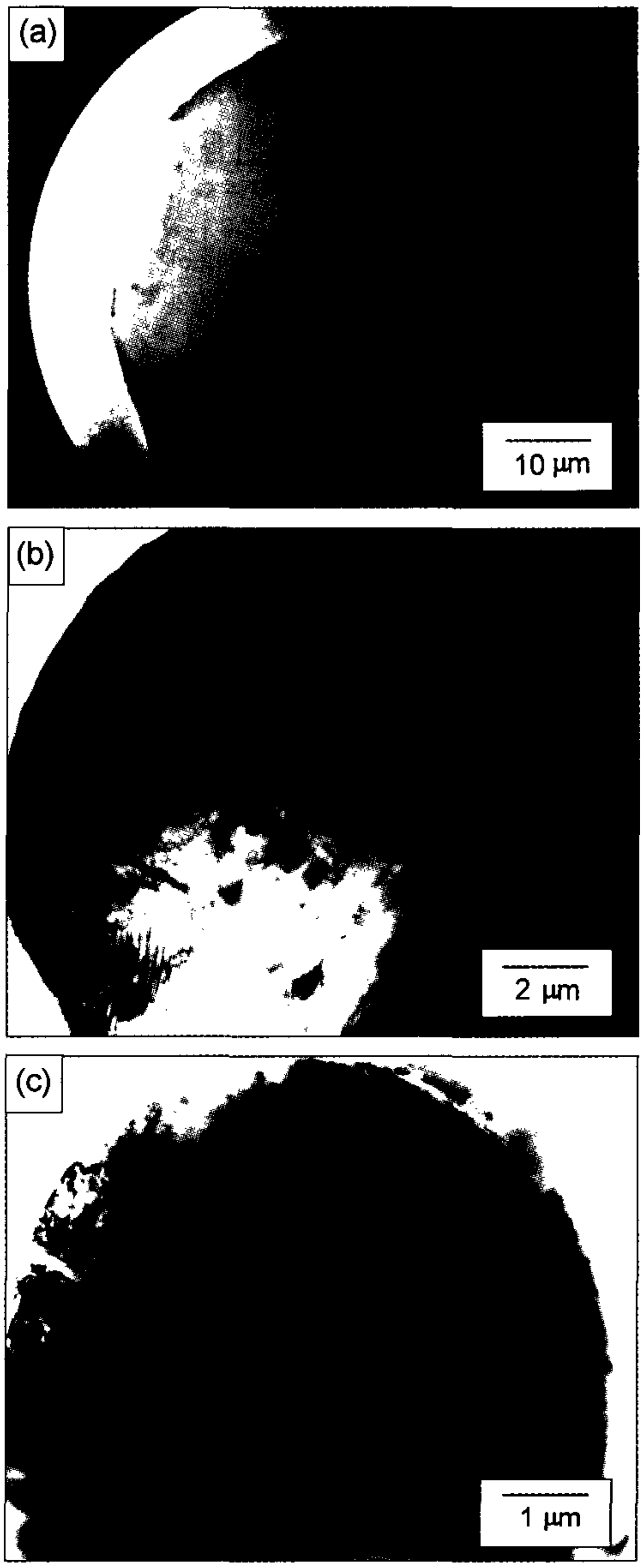

Fig. 2. TEM micrographs of (a) 100 μm diameter powder prepared by mechanical polishing and (b) a 30 μm diameter powder and (c) a powder which is less than 1 μm diameter.

3. Results and Discussion

For the conventionally processed TEM samples the amount of thin area can be quite limited. However there is a large extended thin area on the powders in the size of less than 100 μm diameter range prepared by mechanical polishing, as shown in Fig. 2. Fig. 2(a) is the TEM image of a powder particle larger than 100 μm in diameter, which was polished solely mechanically as a wedge shape using the tripod jig. It is interesting to note that the relatively lager powder is supported and surrounded tightly by the electroplated copper along its edge and about 30 percent area of the powder is transparent even though the sample was not ion milled. When the powder sample was subsequently ion milled

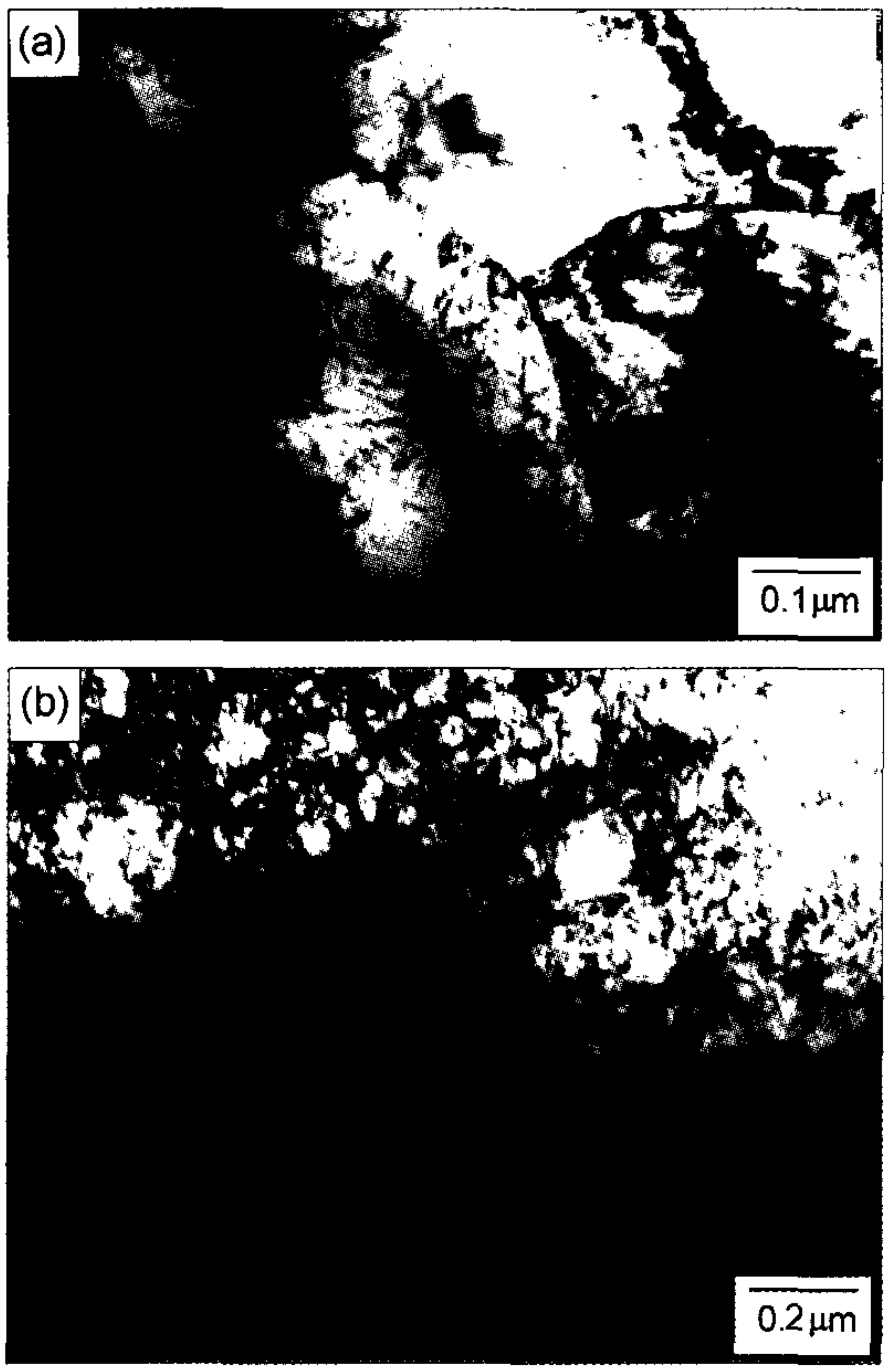

Fig. 3. TEM micrographs of rapidly solidified powders showing (a) grain boundaries and (b) high density of dislocations.

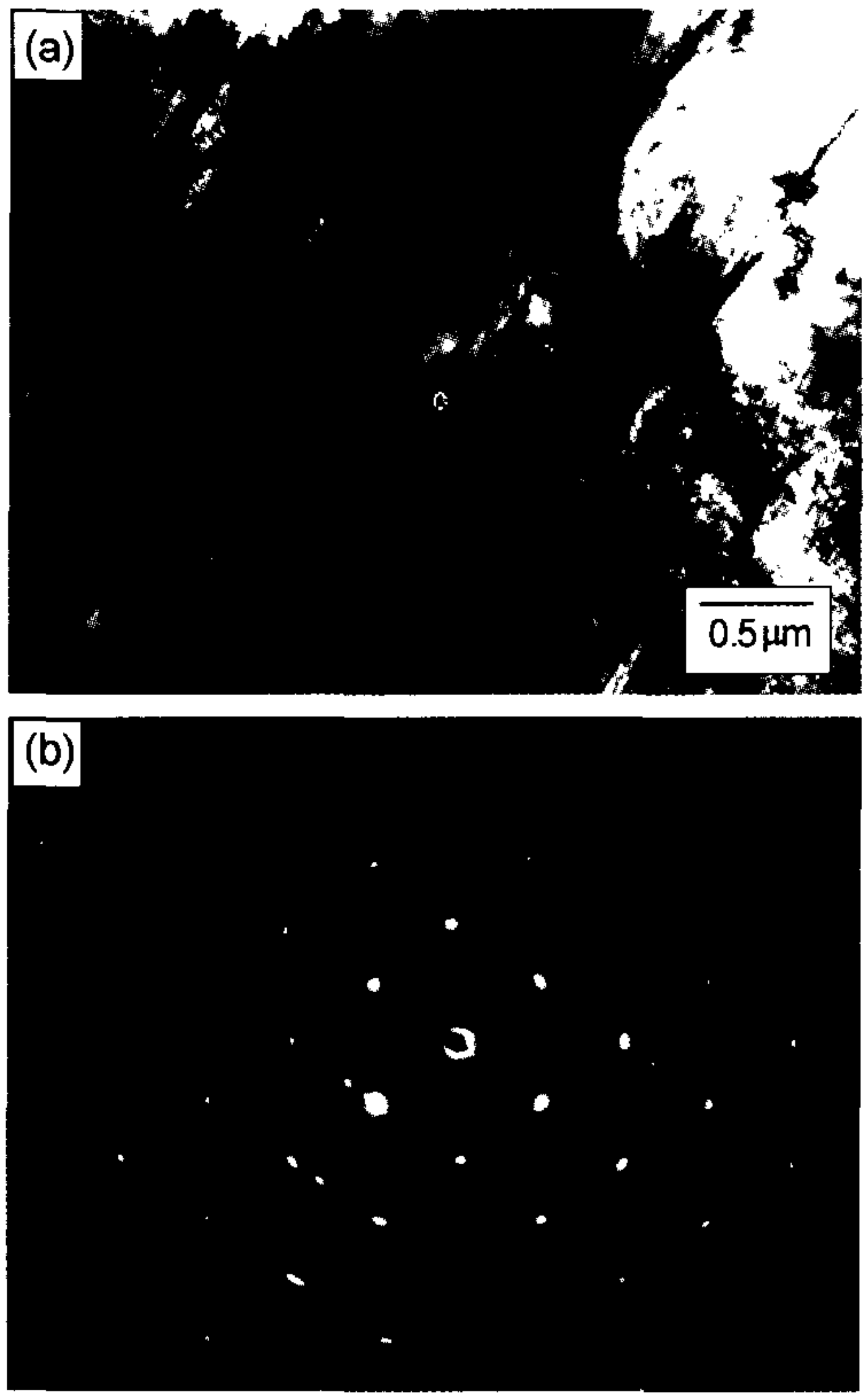

Fig. 4. TEM micrographs of an austenite powder showing (a) twin platelets and (b) a corresponding SADP.

for less than 10 minutes, the large amount of thin area can be examined clearly on TEM as shown in Fig. 2(b). Furthermore, the powder particles less than 1.2 μm in diameter are often entirely electron transparent as shown in Fig. 2(c). According to the analysis of selected area diffraction patterns(SADP) from the powders in the size of 1 and 100 μm diameter range, it is known that most of 304 stainless steel powders rapidly solidified by inert gas atomization solidified as austenite(FCC structure). It was also found that the relatively larger powders(larger than 30 μm diameter) has the high angle grain boundaries as shown in Fig. 3(a) and the powders less than 2 μm diameter such as shown in Fig. 2(c) had the single crystal structures. A significant number of dislocations such as shown in Fig. 3(b) were observed in all powders examined. These microstructural observations are then reflecting that the droplets produced by gas atomization process may undergo high cooling rate during solidification.

The TEM examination on the austenite powders disclosed some interesting microstructure. A lath-like or needle-like shape of planar defects was observed on all powders examined regardless their size. A typical TEM image of this microstructure and a corresponding selected electron diffraction pattern were shown in Fig. 4. The diffraction pattern can be analyzed as the twinned FCC crystal structure that the electron beam direction is $[\bar{1}10]$ and the twin plane (111), the twin direction $[11\bar{2}]$ and extra spots occur at $\frac{1}{3}\vec{g}\langle 111\rangle$ in the reciprocal space as a result of mirror reflections across the (111) plane. The density of this twin band is so high that this feature can be easily observed on the whole area of powder when the sample is tilted with some angles. An illustration is given in Fig. 5 which shows bright field image, dark field image, selected area diffraction pattern and its index. The twinning microstructure is hardly

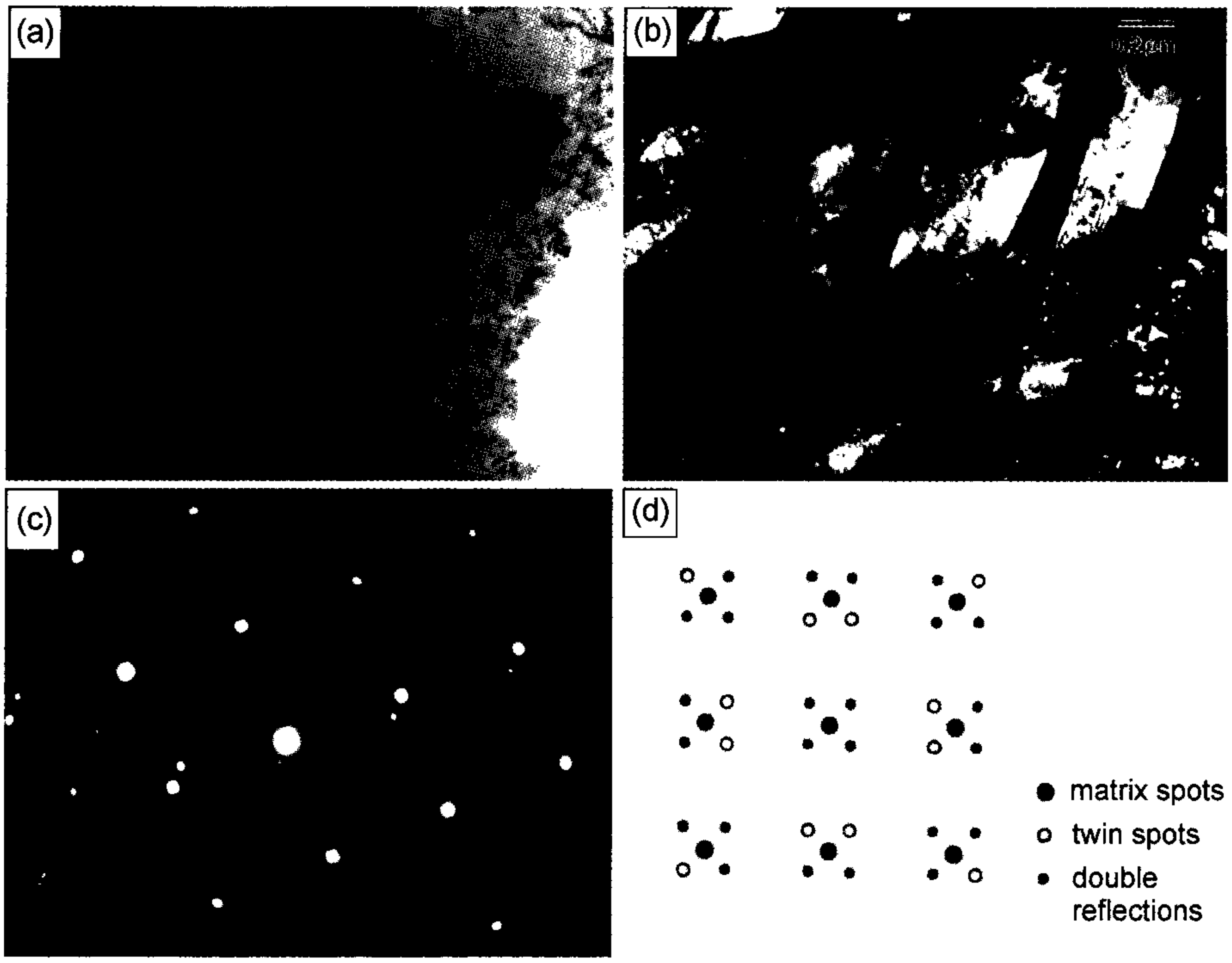

Fig. 5. Bright field image(a) and dark field image(b) of twin and (c) SADP showing double diffraction for {111} twins and (d) its index.

visible in the bright field image in Fig 5(a). However, the diffraction pattern indicates that twins are present in the specimen and a dark field image formed with a twin reflection reveals the distribution of twin platelets as shown in Fig. 5(b). For the diffraction pattern of [001] zone axis as shown in Fig. 5(c), the presence of the twin points reduces the spacing between the planes of reciprocal lattice points parallel to (001) by a factor of three and relatively small lattice rotations cause the appearance of spots due to the twin points in the plane immediately adjacent the (001) plane through the origin. Thus these possible twin spots can be plotted as open circles in Fig. 5(d). Even though it is not common that the twin diffraction pattern like Fig. 5(c) contains extra spots that are located near the transmitted beam. It is possible geometrically that electrons are diffracted in matrix to pass into the twin through its boundary, and diffracted in the twin to pass into the matrix when the twin boundary is not parallel to the electron beam. Thus double diffraction is likely in the vicinity of the twin boundary. In order to consider the effect of double diffraction it can be treated a primary diffracted beam from the matrix as a new source or origin for the twin diffraction pattern. When all extra reflections due to double diffraction are considered, the diffraction pattern of Fig. 5(c) can be indexed as shown in Fig 5(d). This double diffraction spots are showing a good evidence that a high density of twin structure is present in rapidly solidified 304 stainless steel powder.

Recent studies on the tensile behavior of consolidated 304 stainless steel showed that flow stress appeared to be associated with several steps of hardening. According to the microstructural examinations of the consolidated specimen the significant amount of twins are observed and this mechanical behavior is considered as the result of twinning structure[5]. Even though the origin and formation of annealing twins in FCC metals and alloys are well known, the formation of twins may originate in the rapid solidification as observed in this study.

For the smaller powders of less than 2 μm diameter, it

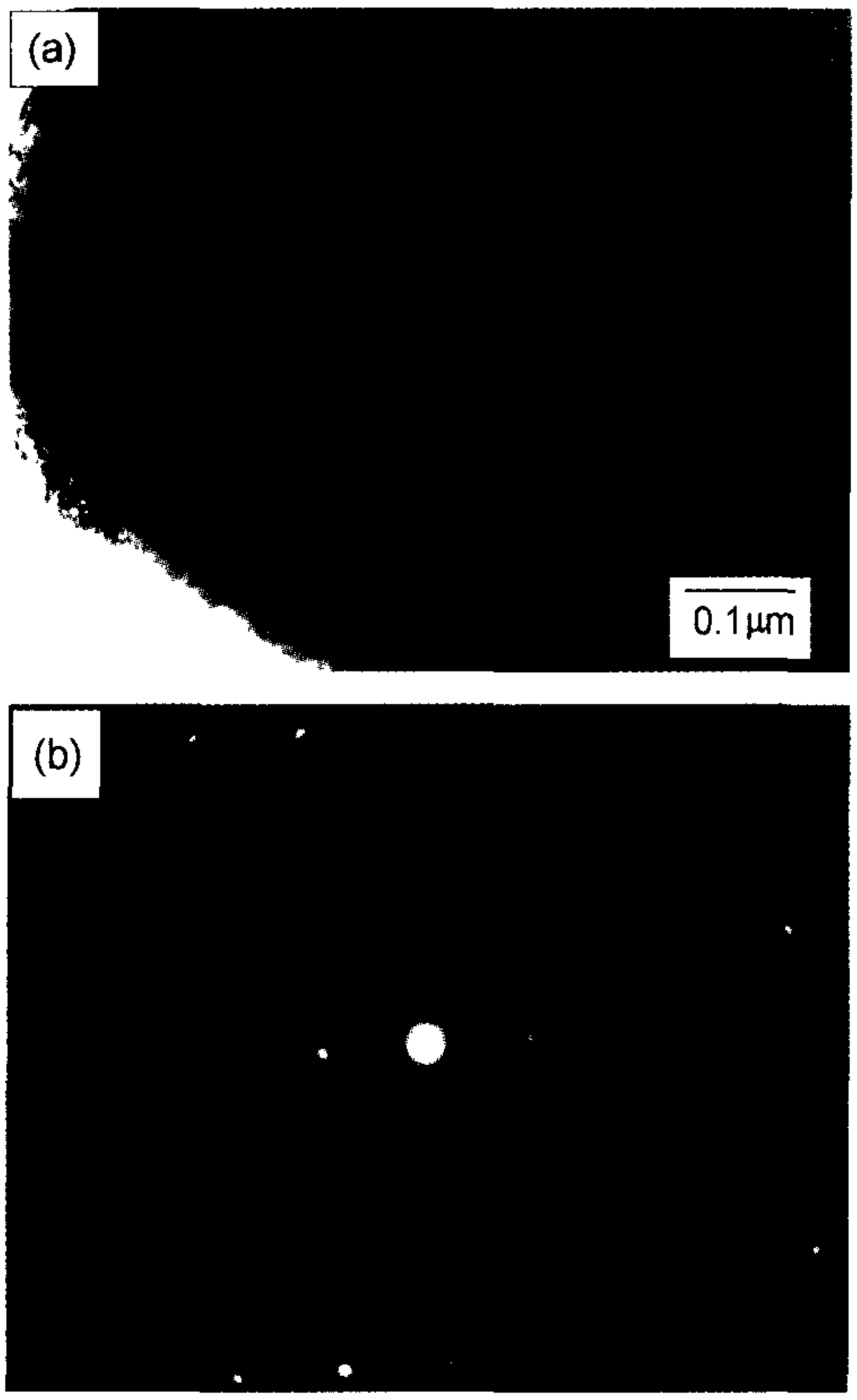

Fig. 6. TEM micrographs of (a) a ferrite powder and (b) a corresponding SADP.

is found that several powders solidified as BCC structure as shown in Fig. 6. The small powders are unlikely to contain potent heterogeneous nucleation sites and therefore the liquid experiences very deep supercooling which favors primary ferrite solidification[6-9]. The larger powders solidifies with much less supercooling as primary austenite. This result is a good agreement with the study of 304 stainless steel powders produced by centrifugal atomization[10].

4. Conclusion

Mechanical polishing using the tripod jig is a useful method to prepare TEM specimens of powder particles because a large extended thin area can be polished at the leading edge of a wedge shape on the powders. Therefore the microstructure of 304 stainless steel powders produced by inert gas atomization process can be investigated on TEM. According to the analysis of SADP from the powders in the size of 1 to 100 μm diameter range, it is known that most of gas atomized powders solidified as austenite. It was also found that several powders of less than 2 μm diameter solidified as BCC structure. It is considered that the small powders are unlikely to contain potent heterogeneous nucleation sites and the liquid droplet experiences a large amount of supercooling which favors primary ferrite solidification. However, the larger powders solidifies with much less supercooling as primary austenite. The most characteristic microstructural feature of rapidly solidified 304 stainless steel powders is the twin band. This needle-like shape of planar defects was observed on all austenite powders examined regardless their size and its density was so high that this feature was observed on the whole area of austenite powders.

Acknowledgment

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