Morphology of graphite in hot-compressed nodular iron

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Bulk material processed by the severe plastic deformation (SPD) technique were found possessing unique physical and mechanical properties [1-3]. SPD material were either nonferrous metal or soft steel. Recently, some researchers attempted to apply this technique to hard-to-work materials [4]. The present authors developed a new SPD process named Cylinder Covered Compression (CCC) and applied it to nodular iron [5]. Our previous work showed that graphite nodules became small with two extended tails along the direction transverse to the compressing force during hot-compression. After severe deformation, a large number of graphite flakes formed a lamellar structure with the metal matrix. This study aimed to build a deformation model of graphite in hot-compressed nodular iron, and then the model was used to explain why graphite area fraction increased on the axial sections of the specimen after more than 80% reduction.

The nodular iron was obtained from the China Railway Shanhaiguan Bridge Co. Ltd, as 22.6 mm thick plates with a chemical composition (mass%) of 3.57C, 2.55Si, 0.22Mn, 0.021P, 0.013S and the balance in Fe.

A new SPD process, named Cylinder Covered Compression (CCC), was developed by the present authors. The schematic illustration of the CCC process is shown in Fig. 1. Specimens, 8 mm in diameter with a 20 mm height, were machined from the cast plates. Cylinders of an 8 mm inner diameter and 10 mm outer diameter were made of medium carbon steel (0.45%C). The specimens enclosed by the cylinders were compressed on a Gleeble 3500 Machine. The details of the thermomechanical treatments are listed in Table I. The 30, 50, 65 and 80% deformed specimens were prepared by one pass of hot-compression. The 80% deformed specimens were cut into pieces, had surface layers ground away, were then stacked, embedded in a cylinder, and hotcompressed again. The 94% deformed specimen was prepared by two passes of about 80% hot-compressions.

TABLE I Summary of the thermomechanical regimes

Reduction (%)	Temperature (°C)	Compressing rate (s^{-1})	Cooling
30	900 ± 2	1×10^{-2}	Air
50	900 ± 2	1×10^{-2}	Air
65	900 ± 2	1×10^{-2}	Air
80	900 ± 2	1×10^{-2}	Air
94	900 ± 2	1×10^{-2}	Air
99.2	900 ± 2	1×10^{-2}	Air

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Figure 1 The schematic illustration of the CCC process.

The 99.2% deformed specimen was prepared by three passes of about 80% hot-compressions.

Each specimen was sectioned axially after each pass of compression and its microstructure was characterized by using optical microscope (OM), image analyzer, and scanning electron microscope (SEM). The metallographic specimens were etched in 2% Nital (2 parts HNO₃ and 98 parts CH₃OH) for 10 s (s). The deepetched specimens for SEM observation were etched in 4% Nital (4 parts HNO₃ and 96 parts CH₃OH) for 1800 s.

All specimens were hot-compressed between flat dies of a Gleeble Machine with mica as the lubricant. The plastic deformation in the specimens was slightly inhomogeneous due to interface friction. Heaviest





Figure 2 Graphite morphology in hot-compressed nodular iron with various reductions: (a) as-cast, (b) 30%, (c) 80%, and (d) 99.2% (not etched).

Figure 3 Microstructure of hot-compressed nodular iron with various reductions: (a) as-cast, (b) 30%, (c) 80%, and (d) 99.2% (etched).

deformation occurred at the center of the specimens. To compare the graphite morphology in different specimens, the examined area was selected at the center of the axial section of the deformed specimens.

Fig. 2 shows the morphology of graphite in hotcompressed nodular iron with various reductions. Fig. 2a represents the graphite morphology of an as-cast specimen. The size of the graphite nodules was inhomogeneous. In 30% deformed specimens, the graphite nodules become saucer-like in shape as shown in Fig. 2b. In this case, the shape of the deformed graphite is very like that in the rolled nodular iron [6]. With the increase in the reduction, the body of the saucer-like graphite shrank and the brim extended along the direction transverse to the compressing force. In the heavily deformed specimens (Fig. 2c), most of graphite nodules had collapsed and a lamellar microstructure of the graphite and steel matrix formed. Further deformation led to the fragmentation of the graphite (Fig. 2d).

Fig. 3 represents the microstructure of the hotcompressed nodular iron corresponding to Fig. 2. All hot-deformed specimens were cooled by air and then the steel matrix was changed into a pearlite structure. In the 30% deformed specimens, only a small part around the graphite was ferrite as shown in Fig. 3b. With the increase in the reduction, ferrite area fraction on the axial section increased. This suggests that hot-deformation leads to the increase of defects in the matrix and then favors the carbon migration from the nearby area to precipitate in the preexisting graphite [6, 7].

Fig. 4 shows SEM micrographs of 99.2% deformed nodular iron (deeply etched). It is clear that the etched areas that enveloped the deformed graphite are the previous ferrite parts in Fig. 3. The brims of the deformed



Figure 4 SEM micrographs of hot-compressed nodular iron (deeply etched).

graphite are normally several microns in thickness and most of them were cracked and fragmented. However, the body parts of the deformed graphite were still kept spheroidal in shape.

One interesting phenomenon observed in Fig. 4b is the aggregation of deformed graphite. In fact, some deformed graphite may start to agglomerate after moderate compression. Basically two combined types of deformed graphite can be sorted out in the present work. One type is that the body of the saucer-like graphite meets the brim of another deformed graphite nodule and merges. Another type is that the bodies of the two saucer-like graphite particles meet and merge. The agglomerated graphite particles will be deformed continuously and merge with other graphite. In heavily deformed specimens, a graphite particle may merge with several other particles as shown in Fig. 4d.

Based on the experimental results above, the deformation model of the graphite in the CCC process can be illustrated by Fig. 5. A graphite nodule becomes saucer-like in shape after a moderate amount of reduction. With the increase of deformation, the body of the saucer-like graphite shrinks and the brim extends. Brim cracking of the graphite may occur at the beginning of deformation. When the amount of reduction reaches 80%, most of the graphite particles are collapsed and the brim of the deformed graphite starts to fragment. Further deformation leads to the decrease in the average diameter of the collapsed graphite. The fragmented graphite flakes flow with the currents of matrix metal and form discontinuous flow lines as shown in Fig. 2c and d.

In highly flat-rolled nodular iron, the graphite may become flaky in shape as well [6, 8]. However, there are some aspects different from that of CCC process. Firstly, graphite nodules are elongated along the rolling direction and form graphite strips in the rolled nodular iron. Secondly, there is no spheroidal body in the center of the deformed graphite. Finally, the deformed graphite is easily fragmented in the rolled nodular iron.



Figure 5 The deformation model of graphite in the CCC process, viewed from the compression direction.

Fig. 6 shows the effect of reduction (%) on graphite area fraction on the axial section of the hot-compressed specimens. It is clear that graphite area fraction varies slightly up to 50% reduction, after which it increases quickly. The graphite area fraction of 99.2% deformed specimens is 18.46%, more than twice that of annealed specimens. The main reason for this phenomenon is the direction deformation in the CCC process. Severe hotcompression makes graphite particles collapse and expand along the direction transverse to the compressing



Figure 6 The effect of reduction (%) on graphite area fraction on the axial section of the hot-compressed specimens.

force, and then more and more graphite can be seen on the axial section of the processed specimens. This structure will lead to the anisotropy of the test materials, especially for damping and mechanical properties.

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References

- 1. R. Z. VALIEV, R. K. ISLAMGALIEV and I. V. ALEXADROV, *Progr. Mater. Sci.* **45** (2000) 103.
- 2. A. BELYAKOV, T. SAKAI, H. MIURA, et al., Acta Mater. 50 (2002) 1547.
- 3. A. BELYAKOV, K. TSUZAKI, H. MIURA, et al., ibid. 51 (2003) 847.
- 4. S. L. SEMIATINA and D. P. DELO, *Mater. Des.* **21** (2000) 311.
- 5. X. ZHAO, T. F. JING, Y. W. GAO, et al., Mater. Lett. in press.
- 6. T. EL-BITAR and E. EL-BANNA, *ibid.* **31** (1997) 145.
- 7. D. A. BARANOV, Liteinoe Proizvod. 2 (2002) 5.
- 8. MUKAE SHIZO, NISHIO KAZUMASA, KATOH MITSUKI, et al., Trans. Jpn. Weld Soc. 23 (1992) 52.

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