A STUDY OF ANTINODULARIZING PROPERTIES OF Pb, Bi, Al AND Ti IN NODULAR CAST IRON BY SPM*

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ABSTRACT

The nodular cast iron samples were prepared with the normal nodularizing process, in which the important antinodularizing elements Pb, Bi and Al were added respectively in order to research the influence of them on the nodularization of the graphites. The nodular graphites were extracted from the nodular cast iron by the electrolytic technique. The distribution of trace elements in nodular and deformed graphite were measured by Scanning Proton Microprobe (SPM). Most of the adding elements Pb, Bi and Al existed in matrix as impurities, a part of them intruded into the nodular graphites. A new suggestion of Ti antinodularizing properties had been proposed. The influence of Pb, Bi and Al on the nodularization was indirectly performed through Ti. Therefore, Ti is one of the most important antinodularizing elements.

Keywords: Nodular graphite Antinodularizing element Elemental distribution SPM

1 INTRODUCTION

Nodular cast iron is a kind of classical metallurgical materials. It is an important industrial material which accounts for over 25 % of ferrous cast irons and is widely used in the automotive industry. Attentions have been paid to the influence of trace elements on the nodularization of graphite in nodular cast iron[1,2]. However, the nodularizing mechanism of graphite is still not yet known very clearly. The antinodularizing properties of some elements in the graphites have not been exactly

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clarified. Normal methods can only give the total contents of trace elements in nodular cast iron. Most of the trace elements exist in the matrix, but they are very low in the nodular graphites. The distribution of element contents above 1000 μg / g can be analysed by Scanning Electron Microprobe (SEM). However the knowledge of contents and distribution of trace elements in nodular graphite is scarce. There are so many trace elements in nodular graphite that the influence of them on the nodularization is complicated. Some information has been obtained with PIXE and SPM in recently years[3,4]. It needs more investigations to get better understanding of trace elements' behaviors.

Two sorts of trace elements are distinguished in nodular cast iron. One has interference to the nodularization of graphites. They are antinodularizing elements, such as Pb, Bi, Al, As and Ti. In general, Pb, Bi and Al are the most important antinodularizing elements. The other belongs to impurity elements. The contents of antinodularizing elements are as low as μg / g in general. The Scanning Proton Microprobe (SPM)[5] has a high spatial resolution and a relative sensitivity of ten μg / g. It can be applied to measure the distribution of trace elements in the nodular graphite. It is a new method to study the properties of antinodularizing elements and nodularizing mechanism further.

2 EXPERIMENTAL

The samples of nodular cast iron were prepared with the normal nodularizing technological process, in which elements Pb, Bi and Al were added respectively to investigate the dependence of the nodularizition of graphite in nodular cast iron on these elements. We measured the mechanical properties and observed the metallography of the samples.

The graphite phase was abstracted from the samples of nodular cast iron using eletrolytic technique. There were many other impurities in the abstracted matters, such as Fe_3C, VC, TiN, MnS, CaO, Al_2O_3 and TiC. These impurities were removed from the abstracted matters by dissolving it in HCl and HCl + H_2O_2 in turn because the graphite has no reaction with acid. After one hour the main impurities would been dissolved. It was checked by Inductively Coupled Plasma (ICP) and PIXE. We obtained pure nodular graphites in this way, a part of them were deformed.

The nodular graphite was deposited into a piece of special plastic slices which resisted high temperature of 300 °C and strong acid. The slices were ground into a thickness of 1 mm, and then lapped till the specimen surface had not any drawn marks. The edges of the graphite balls was very clear when observed with a microscope in 400 times. The polished powders on the surface of the specimen were then removed. The slice was fixed on a Mylar film with a sample frame for SPM analysis. The measurement of distribution of trace elements was performed in SPM laboratory of Lund Institute of Technique in Sweden. The diameter of the beam spot was 5 μm with proton energy of 2.6 MeV and the beam current was 5 nA. A maximum scanning region of 100 μm^2 was obtained in the experimental system. A thick target analysis program without standard (GEO-PIXE) was used to quantitatively analyse the distribution of elements in the graphite.

3 RESULTS AND DISCUSSIONS

The contents of added elements Pb, Al, Bi and Ti in matrix of samples were measured by Inductively Coupled Plasma (ICP) technique. These in nodular graphite of samples were analysed by Proton Induced X-ray Emission (PIXE). The result was shown in Table 1. Pb, Al and Bi is the main antinodularizing elements. All of three elements Pb, Al and Bi strongly affeted the deformed graphite. The content of Pb in the graphite was 187 μg / g when it was 280 μg / g in the matrix of S-Pb sample. The nodularizing percentage 75.9 is lower than that of the certified products (85). The contents of Ti were 240 μg / g in the matrix and 168 μg / g in the graphite of S-Pb sample. The content of Ti in the graphite is higher than that of certified one. The results of S-Al sample are similar to that of S-Pb, but most of Al and Ti exists in the matrix. The added contents of Bi in graphite increased from 20 to 200 μg / g in the matrix of samples S-2Bi and S-3Bi. The contents of Ti increased from 170 μg / g to 200 μg / g in the matrix and from 160 μg / g to 177 μg / g in the graphite separately as well. It indicates that Ti and Bi is a pair of relative elements in the sense of the antinodularization. The higher the content of Bi, the lower the nodularizing percentage is.

Table 1

The contents of added elements Pb, Al, Bi and Ti in matrix and graphite in the samples μg/g

Sample	Nodularizing percentage	Elements added in matrix	Elements added in graphite	Ti in matrix	Ti in graphite
S-Pb	75.9	280	187	240	168
S-Al	71.2	1000	58	180	58
S-2Bi	69.3	20	46	170	160
S-3Bi	53.4	200	74	200	177

The distribution maps of elements show the concentration in various region of graphite. In this paper, we will show the distribution of Fe, Pb, Bi and Ti only. The main composition in matrix is Fe which is higher than 93 %. It is over 2000 μg / g of Fe in graphite. The distribution map of Fe would show the shape of the graphite in SPM analysis. In each figure, the maps marked with "All" are used to exhibit the whole shape of a graphite, which are the distribution maps of all the detected elements in experiments. The mean contents of various elements are listed on the top of each Figure within whole graphite. The darker the grey, the higher the elemental concentration is there.

The shape and elemental distribution of a deformed graphite of S–Pb sample are shown in Fig.1 that the content of Pb is 280 ppm in the matrix. Fig.1a and Fig.1b

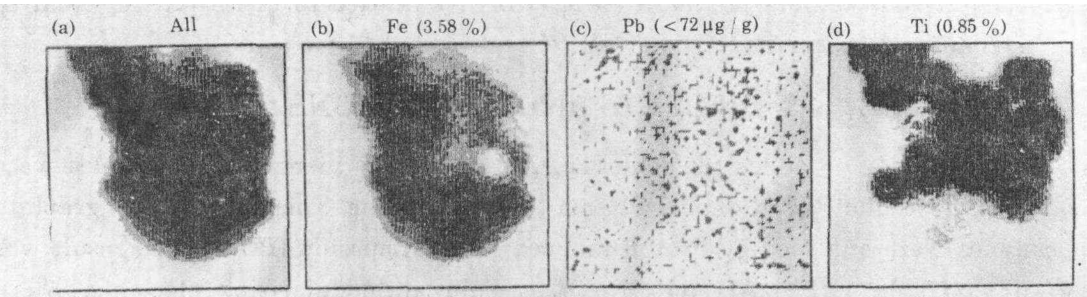

Fig.1 Distribution of elements in deformed graphite within 64 μm×64 μm in S–Pb sample (30 ppm of Pb in matrix)

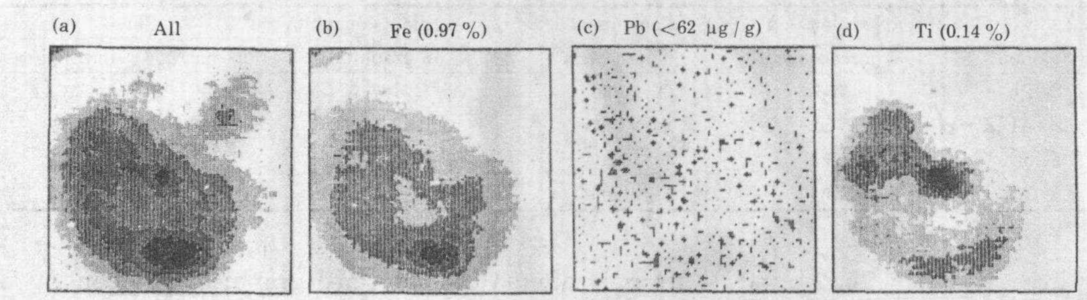

Fig.2 Distribution of elements in nodular graphite within 64 μm×64 μm in S–Pb sample (30 ppm of Pb in matrix)

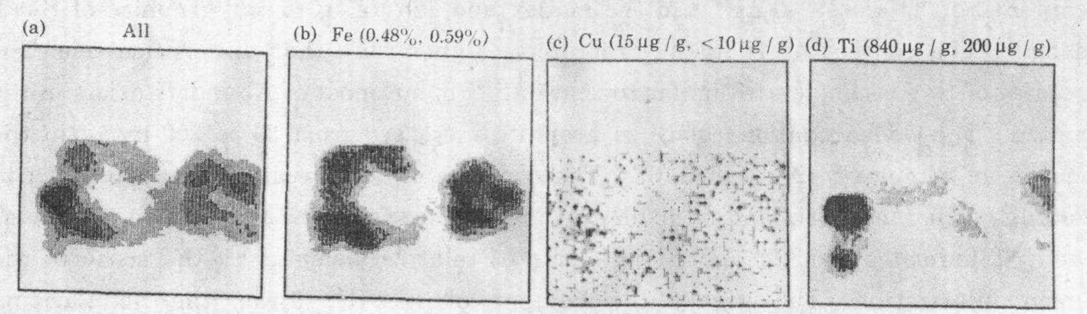

Fig.3 Distribution of elements in deformed and nodular graphite within 96 μm×60 μm in S–Al sample (1000 ppm of Al in matrix)

exhibits the whole shape. The element Pb has intruded into the graphite as shown in Fig.1c. The content of Pb is higher than that of no Pb added samples. The distribution of Ti is not homogeneous in the deformed graphite as shown in Fig.1d. Fig.2a and Fig.2b show another approximate nodular graphite in the same sample. The content of

Pb and Ti in both matrix and graphite are lower than those of the deformed graphite.

The distribution of Al of S-Al sample, which has Al content of 1000 μg / g in the matrix, is not given here because a absorber of Al was used to reduce the intense low energy radiation in the SPM system. Fig.3a and Fig.3b show that there are a deformed

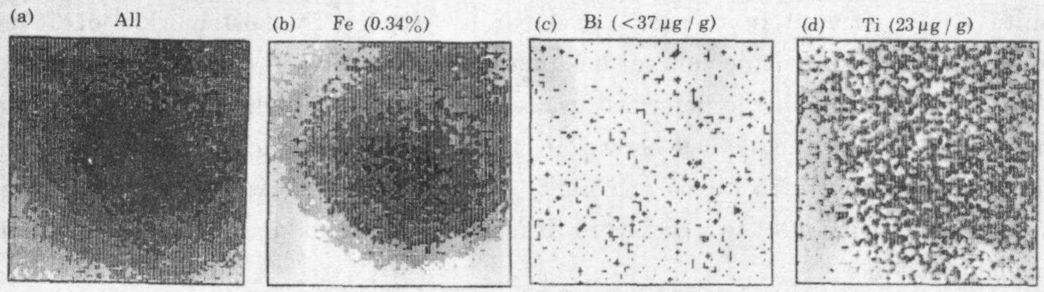

Fig.4 Distribution of elements in nodular graphite within 32 μm×32 μm in S−2Bi sample (20ppm of Bi in matrix)

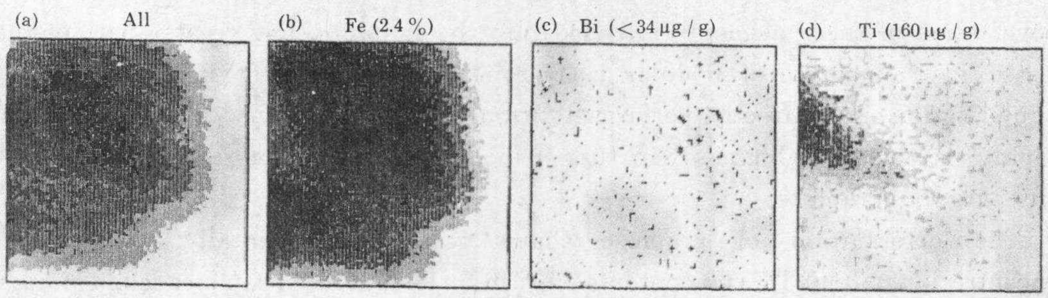

Fig.5 Distribution of elements in deformed graphite within 32 μm×32 μm in S−2Bi sample (20ppm of Bi in matrix)

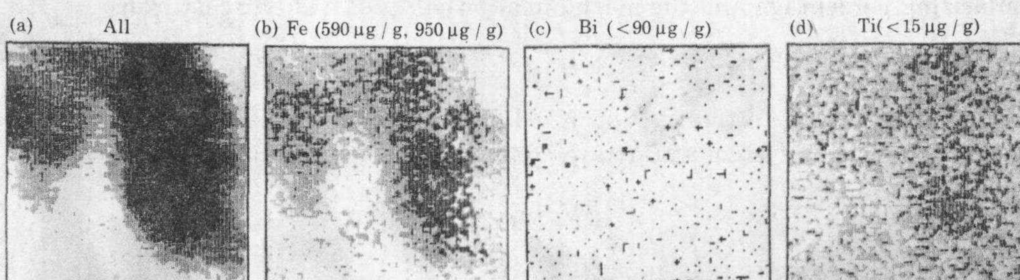

Fig.6 Distribution of elements in nodular and deformed graphite within 64 μm×64 μm in S−3Bi sample (200ppm of Bi in matrix)

graphite in the left and a nodular graphite in the right within a scanning region of 96 μm× 60 μm. The gap of deformed graphite, in Fig.3b, is occupied by rich core of Ti shown as Fig.3d. The content of Ti in the deformed graphite is 840 μg / g and is higher than that in the nodular graphite.

Fig.4 and Fig.5 show the distribution of elements in S-2Bi sample, which the content of added Bi in matrix is 20 μg / g. Fig.4a and Fig.4b exhibits a fine graphite ball. The content of Bi is less than 37 μg / g in this nodular graphite. That is as high as it in the thick target made from the graphite of S-2Bi sample. The content of Ti is obviously reduced. The distribution of Ti is homogeneous as shown in Fig.4d. Fig.5 shows a deformed graphite and the elemental distribution in it. The variation of graphite shape occured in the left as shown in Fig.5b. The distribution of Ti is inhomogeneous there and the content has increased obviously.

The content of Bi in the deformed graphite has increased when it was 200 μg / g in matrix of S-3Bi sample, as shown in Fig.6. There is a nodular graphite and deformed one as shown in Fig.6a and Fig.6b. The content of Ti in Fig.6d is the mean value within the region of two graphites. It is lower in the nodular graphite than that in the deformed one.

4 SUMMARY

According to the distribution and the contents of Pb, Al and Bi in each sample as above, some of these elements intruded into the graphite, but greater part of them existed in the matrix of nodular cast iron. The influence of Pb, Al and Bi on nodularization is indirectly performed through Ti. The content of Ti in deformed graphite is obviously higher than that in nodular one and than the mean value in thick target of graphite.

The deformation of graphite shape occurs coincidentally with a high concentration core of Ti. The contents of Ti in S-Pb and S-Al samples is much higher than that in the matrix. It is suggested that Ti is one of the most important antinodularizing elements. The reduction of the Ti contents will improve the nodularizing percentage and the mechanical property of nodular cast iron.

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