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Corresponding Author: Miss Masuma Mardani,

Corresponding Author's Institution: MISIS

First Author: Masuma Mardani

Order of Authors: Masuma Mardani; Iuliia Fartushna; Alexandra khvan; Vladimir cheverikin; Dmitri Ivanov; alexander kondratiev; Alan Dinsdale

Abstract: Phase equilibria in the Fe-Ce-C system at 1100°C were studied using X-ray diffraction, SEM and electron probe microanalysis. An isothermal section at this temperature was constructed covering the whole concentration range. The stability of the ternary compound  $\Box 1$  at 1100°C, identified previously, was confirmed and its composition determined as 23Fe-29Ce-48C according our data. It is also confirmed that the ternary compound  $\Box 1$  does not have a homogeneity range. The liquid phase is stable at 1100°C in the Fe-Ce-C system. The isothermal section is characterized by five three-phase regions: ( $\gamma$ Fe) + (C) + ( $\alpha$ CeC2), ( $\gamma$ Fe) +  $\Box 1$  + ( $\alpha$ CeC2), ( $\gamma$ Fe) +  $\Box 1$  + (Ce2C3), ( $\alpha$ CeC2) +  $\Box 1$  + (Ce2C3) and L + ( $\gamma$ Fe) + (Ce2C3) plus the corresponding two-phase regions. Dear Editor,

We would like to submit our paper "**Phase equilibria in the Fe-C-Ce system at 1100**°**C**" for publication in the Journal of alloys and compounds. We confirm that this paper represents the authors' own work and that it has not been submitted to any other journal. We also confirm that all authors have read the manuscript and agreed to submit it. We hope that our manuscript will be accepted for the editorial processing. We are looking forward for your reply. Best regards,

M. Mardani, I. Fartushna, A. Khvan, V. Cheverikin, D. Ivanov, A. Kondratiev, A. Dinsdale

Phase equilibria in the Fe-C-Ce system at 1100°C were studied using X-ray diffraction, SEM and electron probe microanalysis. An isothermal section at this temperature was constructed covering the whole concentration range.

The authors of the paper confirm that:

1. This manuscript is our original work and has not been published nor has it been submitted simultaneously elsewhere.

2. That all authors have checked the manuscript and have agreed to the submission.

Dear Dmitry G Eskin,

The authors are thankful to the reviewers for their thorough reviews and useful comments. The required corrections have been made in the revised paper. Review of the paper "Phase equilibria in the Fe-Ce-C system at 1100°C" (JALCOM-D-17-08498)

Reviewers' comments:

1. The composition found for the ternary phase should be included in the abstract.

The composition of the ternary phase is included.

2. The first sentence in the Introduction ("The introduction of rare-earth metals (R)...") makes no sense. It should be reformulated or deleted.

The first sentence has been deleted.

3. Section 3.2, last sentence: Does reference 14 contain a statement about CeC6?

The ref. has been corrected.

4. I could not find Ref. 27 in the text.

I have added a reference to ref 27 in the text.

#### Reviewer #2:

1. Cerium can react with O2 in the air slowly. It is recommended that the authors re-analyze the samples including O2.

The samples have been melted in an arc furnace and kept in a protective atmosphere. A microstructure analysis of the samples was carried out directly after polishing the samples, so the samples did not have enough time to react with  $O_2$ . In addition, we did not see any oxide phases on the microstructures or in the XRD results. This Explanation was added to section 2.1.

2. The authors summarize the alloys data in Table 1. But the phase compositions were not given. On the other hand, from Fig.3a, it is not so convincing to conclude the existence of the CeC2 phase. Considering these facts, the experimental results are quite questionable. The authors mentioned that EPMA determined carbon content is not reliable. However, the phase composition determination is critical for the construction of the Fe-Ce-C isothermal section at 1373 K. Thus, the authors should consider using other techniques to determine the phase compositions. By the way, the term" phase composition" used in this paper is wrong. The correct term is "phase identification".

We changed the term "phase composition" to "phase identification" in the manuscript as well as the Table 1 and added EPMA results.

Indeed, the amount of the CeC2 phase in the sample Ce4Fe4C7 is very low, so the peaks corresponding to this phase are barely noticeable. However, this phase is clearly visible on the microstructure of this sample. Therefore, we presented an additional X-ray diffraction pattern of the sample Ce3Fe5C6 from the same three-phase region, on which peaks corresponding to the CeC2 phase are clearly visible.

Unfortunately, the EDX analysis has its limitations. In order to decrease errors, which are inevitable during the measurement of carbide compositions, and increase the precision of the measurement, further calibrations using the carbides Fe3C, Ce2C3 and CeC2 were carried out. However, the error of the carbon determination nevertheless exceeds the error of metal determination. But the fact that none of the phases show any variation of the lattice parameters indicates that the phases don't have any solubility of a third component. Therefore, the location of the three-phase regions cannot differ substantially from that shown in Fig. 1. In addition, the position of the three-phase regions was also determined by the ratio of phases in the two and three-phase alloys.

3. Page 3, last 4 line "Cooling of the alloys was performed with the furnace turned off". The reviewer really has concerns about this point. According to this sentence, furnace cooling was used. Please check carefully if this is true.

It has been corrected.

# Phase equilibria in the Fe-Ce-C system at 1100°C

M. Mardani<sup>1</sup>, I. Fartushna<sup>1</sup>, A. Khvan<sup>1</sup>, V. Cheverikin<sup>1</sup>, D. Ivanov<sup>1</sup>, A. Kondratiev<sup>1</sup>,

A. Dinsdale<sup>2,3</sup>

<sup>1</sup> Thermochemistry of Materials Scientific Research Centre, NUST MISIS, Leninsky prosp. 4, 119049 Moscow, Russia <sup>2</sup> Hampton Thermodynamics Ltd, UK <sup>3</sup> BCAST, Brunel University London, Uxbridge, UK, UB8 2AD

# Abstract

Phase equilibria in the Fe-Ce-C system at 1100°C were studied using X-ray diffraction, SEM and electron probe microanalysis. An isothermal section at this temperature was constructed covering the whole concentration range. The stability of the ternary compound  $\tau_1$  at 1100°C, identified previously, was confirmed and its composition determined as 23Fe-29Ce-48C according our data. It is also confirmed that the ternary compound  $\tau_1$  does not have a homogeneity range. The liquid phase is stable at 1100°C in the Fe-Ce-C system. The isothermal section is characterized by five three-phase regions: ( $\gamma$ Fe) + (C) + ( $\alpha$ CeC<sub>2</sub>), ( $\gamma$ Fe) +  $\tau_1$  + ( $\alpha$ CeC<sub>2</sub>), ( $\gamma$ Fe) +  $\tau_1$  + (Ce<sub>2</sub>C<sub>3</sub>), ( $\alpha$ CeC<sub>2</sub>) +  $\tau_1$  + (Ce<sub>2</sub>C<sub>3</sub>) and L + ( $\gamma$ Fe) + (Ce<sub>2</sub>C<sub>3</sub>) plus the corresponding two-phase regions.

*Keywords:* Phase diagram, ternary compound, isothermal section, Fe-Ce-C, rare earth metal, phase equilibria, lanthanides

\*Corresponding author.

E-mail address: <u>masuma.mardani@gmail.com</u>

### **1. Introduction**

Rare-earth metals can be used to clean steels [1-3] and act as an inoculant in steels to form vermicular or nodular graphite. However, the mechanism of the influence of rare-earth additions on the properties of the steel is still not clear. Thus, an investigation of the phase equilibria in the Fe-Ce-C system is of great practical importance for the steel industry. In [4-7] it was reported that ternary compounds forming in Fe-R-C systems have been proposed as potential magnet materials.

Phase equilibria in the Fe-Ce-C system were studied experimentally by [8, 9]. Park et al. [8] investigated the Fe-Ce-C system in the composition range up to 40 at. % Ce and 60 at. % C on the basis of light microscopy and x-ray analysis of as-cast and annealed samples and provided a liquidus projection and isothermal sections at 800°C and 950°C, which were accepted later in the review of Raghavan [10] but redrawn to be compatible with the accepted binary data. Recently, we studied the phase equilibria in this system on crystallization using DTA, X-ray diffraction, SEM and electron probe microanalysis [9]. Liquidus and solidus projections for this system over the whole concentration range were constructed. This work is focused on an experimental study, using SEM, EPMA and X-ray diffraction, of phase equilibria in the Fe-Ce-C system at temperature of 1100°C.

## **1.2 Literature survey**

### **Binary** systems

The phase diagrams for the Ce-Fe and C-Fe binary systems from assessments [11] and [12], respectively are accepted and are in good agreement with all available experimental data. There are two stable intermetallic compounds in the Ce-Fe system, Ce<sub>2</sub>Fe<sub>17</sub> and CeFe<sub>2</sub> (C15 Laves phase). The compound Ce<sub>2</sub>Fe<sub>17</sub> exists in two modifications, the temperature of the transformation is not known [13]. The high temperature modification ( $\beta$ Ce<sub>2</sub>Fe<sub>17</sub>) has a rhombohedral Th<sub>2</sub>Zn<sub>17</sub>-type, while the low temperature modification ( $\alpha$ Ce<sub>2</sub>Fe<sub>17</sub>) has a hexagonal Th<sub>2</sub>Ni<sub>17</sub>-type [13]. The solubilities of Ce in ( $\delta$ Fe), ( $\gamma$ Fe) and ( $\alpha$ Fe) and Fe in ( $\delta$ Ce) and ( $\gamma$ Ce) are negligible. The assessment of data for the C-Fe system, carried out by Gustafson [12], is still widely used and is accepted in this work. Recently Naraghi et al. [14] also described this system; no significant changes in the phase equilibria at high temperature were made.

The binary C-Ce system adopted is based on the experimental work of [15]. The assessment [16] for this system did not include these experimental data. Two carbides form in this system, Ce<sub>2</sub>C<sub>3</sub> and CeC<sub>2</sub>. The CeC<sub>2</sub> carbide melts congruently at 2340°C and has two modifications. The high temperature modification ( $\beta$ CeC<sub>2</sub>) has a cubic CaF<sub>2</sub>-type structure, while the low temperature modification ( $\alpha$ CeC<sub>2</sub>) has a tetragonal structure of the CaC<sub>2</sub> type. The phase ( $\beta$ CeC<sub>2</sub>) has narrow homogeneity region. The solubilities of C in ( $\delta$ Ce) and ( $\gamma$ Ce) are ~10 at.%.

Bonhomme and Gosselin [17] also identified a compound  $CeC_6$ . However, the assessments [16, 18, 19] and the experimental work [8, 9, 15] did not include this compound.

#### Ternary intermetallic compounds

Park et al. [8] observed two intermetallic compounds  $Ce_4Fe_4C_7$  and  $Ce_2Fe_2C_3$  which form through solid state reactions. The formation temperature of the ternary compound  $Ce_4Fe_4C_7$  is above 950°C while the formation of the compound  $Ce_2Fe_2C_3$  has been reported to be between 950 and 800°C [8]. A compound  $Ce_4Fe_4C_7$  ( $\tau_1$ ) has also been reported by [20] although its composition is not known exactly. Mooij et al. [21] estimated this composition to be close to  $R_3Fe_5C_6$  (R = Ce, Pr, Nd, Sm and Gd) and indicated that  $R_3Fe_5C_6$  is a refined composition of the previously reported compound  $R_4Fe_4C_7$  (R = Ce, Gd) [8, 22].

The compounds reported with the tentative compositions  $Ce_{10}Fe_3C_{17}$  [23] and  $Ce_2Fe_2C_3$ [8] seem to be identical with the carbide  $Ce_{3.67}FeC_6$  [24]. Recently , in [25] the  $La_{3.67}[Fe(C_2)_3]$  compound with a structure  $P6_3/m$  was reinvestigated.

The compounds reported with the compositions  $R_3Fe_{20}C$  (R = La [23], Gd [22]) and  $R_{14}Fe_{78}C_8$  [26] seem to be identical with the composition  $R_2Fe_{14}C$  (Nd<sub>2</sub>Fe<sub>14</sub>B-structure type, *P*4<sub>2</sub>/*mnm*) [27]. In [6] it was demonstrated that Ce<sub>2</sub>Fe<sub>14</sub>C can be formed by the rapid solidification technique of melt spinning together with a comparatively brief heat treatment at an appropriate temperature.

### **2** Experimental

#### **2.1 Sample preparation**

The alloys were melted in an arc-furnace with a tungsten electrode on a water-cooled copper hearth in an Ar atmosphere 99.998% pure (pressure 80 kPa), purified by a Ti-melt with the purity of the starting materials, Fe–99.99%, C–99.8% and Ce–99.8%. To achieve homogeneity, the buttons were turned over and remelted six times. The weights of the ingots were typically 3 g. The weight losses did not exceed 0.1 %. So, generally the composition of the samples was taken according to their nominal composition.

The alloys were subjected to homogenization heat treatment at 1100°C for 24-85 hours. The annealing was performed in a tube furnace (Nabertherm RHTV 120/300/1700) with a temperature accuracy of  $\pm 3$ °C. Annealing took place in an argon atmosphere 99.998% pure. The samples were placed in an Al<sub>2</sub>O<sub>3</sub> crucible. The samples were subjected to furnace cooling.

In order to preserve the samples, that show a tendency to disintegrate, the samples were kept in protective atmosphere and microstructure analysis of the samples was carried out directly after polishing the samples, so the samples did not have enough time to react with  $O_2$ . In addition, we did not see any oxide phases on the microstructures or in the XRD results.

X-ray diffraction (XRD), optical (OM), scanning electron microscopy (SEM), and electron microprobe analysis (EPMA) were used for investigation of this system.

### 2.2 Microstructure analysis

Metallographic specimens for microscopic examination were prepared by a Struers Labopol-5 machine. Firstly, the samples were ground using SiC-paper and then were polished with diamond discs with grain sizes of 9, 6, 3 and 1 microns. A diamond suspension was sprayed at regular intervals during the preparation. The microstructure of the alloys was examined using optical microscopy (OM) (Olympus-GX71F-5) and scanning electron microscopy (SEM) using a TESCAN VEGA LMH microscope with a  $LaB_6$  cathode and an energy dispersive X-ray microanalysis system - Oxford Instruments Advanced AZtecEnergy. Both backscattered electron and secondary electron imaging were used in the analysis. An electron microprobe analysis (EPMA) was carried out on all phases using a four-crystal wave spectrometer (the analysed particle size was larger than 2 µm). The acceleration voltage used for the EPMA was 20 kV. The error of measurement in determining the concentration of elements using X-ray analysis was 0.2 wt.%. The detector was calibrated using standard samples of Co (99.99%), Cu (99.999%), Ni (99.99%), Mn (99.99%), Fe (99.999%), CeO<sub>2</sub> (99.99%), C (99.99%) and Cr (99.99%) from Oxford Instruments. In order to minimize errors, which are inevitable during measurement of carbide compositions, and increase the precision of the measurements, further calibrations using the carbides  $Fe_3C$ ,  $Ce_2C_3$  and  $CeC_2$  were carried out.

## 2.3 X-ray diffraction analysis

X-ray diffraction (XRD) analysis was applied to determine the phase identification of the alloys using CuK $\alpha$  radiation operated at a voltage of 40 kV, a current of 40 mA and filtered with a Ni-crystal monochromator. XRD measurements were performed using a multipurpose X-ray diffractometer (Bruker-AXS D8 Discover). A parallel beam with a divergence of 0.03° is formed using the mirror of Gobel. The reflected intensity of the beam was measured using LYNXEYE position sensitive detector (angular resolution of 0.015°). Indexing of the reflections was performed with the WinXPOW and PowderCell software products.

#### 3. Results and discussion

The alloys were studied after annealing at 1100°C for 24-85 hours. A complete list of investigated samples in this system, as well as the phase identification of the alloys and EPMA data is given in Table 1. An isothermal section of the Fe-Ce-C system at this temperature was constructed and shown in Fig. 1. The microstructures of some of the annealed samples are presented in Fig. 2. The lattice parameters of the solid phases are shown in Table 2.

#### 3.1 The ternary compound $\tau_1$

The ternary compound  $\tau_1$  (Nd<sub>3</sub>Fe<sub>5</sub>C<sub>6</sub>-type structure, *P*-4*m*2, *a* = 7.231, *c* = 3.192 Å [21]), which forms in solid state by peritectoid reaction ( $\gamma$ Fe) +  $\beta$ CeC<sub>2</sub> + Ce<sub>2</sub>C<sub>3</sub>  $\rightleftharpoons \tau_1$ , was found to exist at a temperature of 1100°C. The ternary compound  $\tau_1$  at 1100°C does not show any variation of the lattice parameters on changes of composition, which indicates that  $\tau_1$  does not have a homogeneity range (Table 2).

The composition of  $\tau_1$  is not exactly known. The correct composition of the  $\tau_1$ -phase can be determined from EPMA. Unfortunately the quality of EPMA is moderate, and considerably exceeds the error of the carbon content determination, that is why the ratio of metallic contents Ce/Fe only was determined. Samples ## 8, 9, 12-16 were investigated using electron probe microanalysis and it was found that these alloys contain the ternary compound  $\tau_1$ , with a Ce/Fe ratio of 56/44. From the results of SEM, EPMA and XRD on the alloys #8 (Ce<sub>4</sub>Fe<sub>4</sub>C<sub>7</sub>) and #12 (Ce<sub>3</sub>Fe<sub>5</sub>C<sub>6</sub>), and the observation of additional phases ( $\gamma$ Fe) and  $\alpha$ CeC<sub>2</sub> in the sample #12 with the overall composition of (Ce<sub>3</sub>Fe<sub>5</sub>C<sub>6</sub>), it is confirmed that the composition of the  $\tau_1$ -phase is closer to the composition of Ce<sub>4</sub>Fe<sub>4</sub>C<sub>7</sub>, than to Ce<sub>3</sub>Fe<sub>5</sub>C<sub>6</sub> (Figs. 2 *a*, *b*, 3 *a*, *b*). In addition, the presence of phases ( $\gamma$ Fe) and  $\alpha$ CeC<sub>2</sub> in the alloy #8 indicate that the Ce content in the  $\tau_1$ -phase is a little higher than given by the stoichiometry Ce<sub>4</sub>Fe<sub>4</sub>C<sub>7</sub>, while the Fe content in this phase is slightly lower. This was also observed in [20], the atom ratio in  $\tau_1$ -phase is Ce:Fe:C = 4.4:4:7.2. Thus, according to our data the composition of  $\tau_1$  is 23Fe-29Ce-48C.

The crystal structure of the ternary compound  $\tau_1$  is not completely resolved. Park et al. [8] determined that the compound Ce<sub>4</sub>Fe<sub>4</sub>C<sub>7</sub> ( $\tau_1$ ) had a primitive tetragonal structure ( $tP^*$ , a = 7.22, c = 5.82 Å). Mooij et al. [21] attempted to find a crystal structure for this phase and provide a possible model for filling a unit cell with heavy atoms on the basis of a Nd<sub>3</sub>Fe<sub>5</sub>C<sub>6</sub>-type structure, (*P*-4*m*2, a = 7.231, c = 3.192 Å). However, the crystal structure has not been resolved completely yet. It should be noted that some unexplained reflection lines are present in the X-ray patterns of the samples ##8, 11-13 (Fig. 3 *a*).

In order to check the existence of other ternary compounds in the Fe-Ce-C system at 1100°C, we prepared several alloys with the compositions of possible ternary compounds which had been found in Fe-R-C systems.

The ternary compound  $Ce_{3.67}FeC_6$  ( $\tau_2$ ) (# 16) [24, 25] which had been identified by [8] with the tentative composition  $Ce_2Fe_2C_3$  (# 9) was not observed by us in the annealed alloys at 1100°C. This confirms that the ternary compound  $\tau_2$  forms through a peritectoid reaction ( $\gamma Fe$ ) +  $Ce_2C_3 \rightleftharpoons \tau_2$  at a temperature between 950 and 800°C [8].

The ternary compound with the composition of RFeC<sub>2</sub> (ScCoC<sub>2</sub>-type structure, *tP*8-*P4/nmm* and CeNiC<sub>2</sub>-type structure, *oS*8-*Amm2*) which had been found in systems with R = Sc, Y, Sm, Gd-Er and Lu [21, 29-32], was not found in Fe-Ce-C system. However, the general view of microstructure of alloy # 11 with composition CeFeC<sub>2</sub> reveals a small amount of the  $\tau_1$ -phase and existence of ( $\gamma$ Fe) and  $\alpha$ CeC<sub>2</sub>. This gives us reason to believe that the composition of alloy # 11 is almost on the boundary tie-line ( $\gamma$ Fe) +  $\alpha$ CeC<sub>2</sub>. Alloy #10 with composition Ce<sub>3</sub>Fe<sub>2</sub>C<sub>6</sub> has been shown to contain two-phases ( $\gamma$ Fe) +  $\alpha$ CeC<sub>2</sub>. It should be noted that the ternary compound R<sub>3</sub>Fe<sub>2</sub>C<sub>6</sub>, of unknown crystal structure, was found only in the system with Dy at 800°C [28]. In [33] the existence of a compound close to the composition R<sub>2</sub>FeC<sub>4</sub> with an orthorhombic crystal structure (Er<sub>2</sub>FeC<sub>4</sub>-type structure, *oI28-Ibam*) for R = Y, Tb-Lu was reported, we did not observe this compound in Fe-Ce-C system at 1100°C.

Another ternary compound with composition  $R_{11}Fe_{12}C_{18}$ , which was found in the system with Sm (Th<sub>11</sub>Ru<sub>12</sub>C<sub>18</sub>-type structure, *cI82-I-43m*) [34], does not form at 1100°C in the Fe-Ce-C system, based on the SEM, EPMA and XRD results on alloy # 13 with the composition  $Ce_{11}Fe_{12}C_{18}$ . This alloy contains the three phases  $\tau_1 + (\gamma Fe) + Ce_2C_3$ , the amount of  $\tau_1$ -phase in this alloy being very high (Figs. 2 *c*, 3 c), and consequently, the alloy is located close to the composition of the ternary compound  $\tau_1$ . This further proves that the composition of  $\tau_1$ -phase is closer to the composition of  $Ce_4Fe_4C_7$ , than to  $Ce_3Fe_5C_6$ .

The novel intermediate ternary phases  $R_{15}Fe_8C_{25}$  (R = Y, Dy, Ho, Er) (Er<sub>15</sub>Fe\_8C<sub>25</sub>-type structure, *hP49-P321*) have been identified by [28, 35, 36]. The alloy # 14 with the composition  $Ce_{15}Fe_8C_{25}$  contains only phases  $\tau_1 + Ce_2C_3 + \alpha CeC_2$  (Fig. 2 *d*). In [36] the ternary phase  $R_{5.64}Fe_2C_9$  (R = Y, Gd, Tb, Dy) with an orthorhombic crystal structure *Pnma* also has been reported. We did not observe this phase in Fe-Ce-C system. It is most likely that these compounds form only in R-Fe-C systems with heavy rare-earth metals.

Therefore, no other ternary compounds, besides  $\tau_1$ , were identified in the Fe-Ce-C system at a temperature of 1100°C

### 3.2 Phase equilibria

At the temperature of 1100°C,  $\tau_1$  is in equilibrium with the phases ( $\gamma$ Fe), ( $\alpha$ CeC<sub>2</sub>) and (Ce<sub>2</sub>C<sub>3</sub>) forming three, three-phase fields, namely ( $\gamma$ Fe) +  $\tau_1$  + ( $\alpha$ CeC<sub>2</sub>), ( $\gamma$ Fe) +  $\tau_1$  + (Ce<sub>2</sub>C<sub>3</sub>), ( $\alpha$ CeC<sub>2</sub>) +  $\tau_1$  + (Ce<sub>2</sub>C<sub>3</sub>), and the corresponding two-phase regions. In addition, two more three phase regions, ( $\gamma$ Fe) + (C) + ( $\alpha$ CeC<sub>2</sub>) and L + ( $\gamma$ Fe) + (Ce<sub>2</sub>C<sub>3</sub>) are present in the phase diagram at this temperature (Fig. 1).

The microstructure results of the alloys ## 8, 11 and 12 clearly show three distinct phases: black grains, a grey matrix and dark-grey grains (Fig. 2 *a*, *b*) which correspond to ( $\gamma$ Fe),

 $\tau_1$  and ( $\alpha \text{CeC}_2$ ), respectively and this is confirmed by XRD data (Figs. 3 *a*, *b*). Therefore these samples are located in the ( $\gamma \text{Fe}$ ) +  $\tau_1$  + ( $\alpha \text{CeC}_2$ ) three-phase region

In the SEM micrograph and the X-Ray of samples #9 and #13, three phases,  $\tau_1$  (lightgrey matrix) + ( $\gamma$ Fe) (grey grains) + (Ce<sub>2</sub>C<sub>3</sub>) (white grains), are well distinguished (Figs. 1, 2 *c*, 3 *c*, Tables 1-2). The amount of the  $\tau_1$ -phase in the alloy #13 is very high and, consequently, the alloy is located close to the composition of the ternary compound  $\tau_1$ . On the other hand the amount of Ce<sub>2</sub>C<sub>3</sub> is very low and, therefore, the composition of this alloy is almost on the boundary tie-line  $\tau_1 + (\gamma$ Fe).

Moreover, another three-phase region involving the  $\tau_1$ -phase is formed in the Fe-Ce-C system at 1100°C. It was determined with SEM, EMPA and XRD of the three-phase alloys ## 14-16, which contain the three phases ( $\alpha$ CeC<sub>2</sub>) (grey grains)+  $\tau_1$  (light-grey matrix) + (Ce<sub>2</sub>C<sub>3</sub>) (white grains) (Tables 1-2, Figs. 1, 2 *d*).

The location of the three-phase region  $(\gamma Fe) + (C) + (\alpha CeC_2)$  was established based on the results for alloys #4, 5 and 7 with three phases in equilibrium (Figs. 2 *e*, 3 *d*) and alloys ##1, 3 and 10 with the two-phases  $(\gamma Fe) + (\alpha CeC_2)$  in equilibrium (Tables 1-2, Fig. 1).

The location of the liquid phase was determined by the construction and processing of a series of intersecting isopleths. A three-phase region involving the liquid phase,  $L + (\gamma Fe) + (Ce_2C_3)$ , is present. The isopleths were used to refine the location of this three-phase region containing the liquid-phase.

No binary intermetallic phases have appreciable solubility of the third component. The  $CeC_6$  compound reported in [17] was not found by us in the ternary Fe-Ce-C system at 1100°C.

### 4. Conclusions

Phase equilibria in the Fe-Ce-C system at  $1100^{\circ}$ C were studied over the whole concentration region. An isothermal section at this temperature was constructed. The ternary compound  $\tau_1$  is stable at this temperature and is in equilibrium with the phases ( $\gamma$ Fe), ( $\alpha$ CeC<sub>2</sub>) and (Ce<sub>2</sub>C<sub>3</sub>). No binary intermetallic phases have appreciable solubility of the third component.

## Acknowledgments:

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## **Figure captions**

Fig. 1. The isothermal section at 1100°C of the Fe-Ce-C system:  $\bullet$  – two-phase samples;  $\bullet$  – three-phase samples.

Fig. 2. Microstructure of annealed samples at 1100°C of alloys in the Fe-Ce-C system:

$$a - Ce_4Fe_4C_7$$
 (#8), ×2000,  $\tau_1 + (\gamma Fe) + (\alpha CeC_2)$ ;

- $b Ce_3Fe_5C_6$  (#12), ×2000,  $\tau_1 + (\gamma Fe) + (\alpha CeC_2)$ ;
- $c-Ce_{11}Fe_{12}C_{18} \ (\#13), \times 2000, \ \tau_1+(\gamma Fe)+(Ce_2C_3);$
- $d Ce_{15}Fe_8C_{25} \ (\#14), \times 2000, \ \tau_1 + (\alpha CeC_2) + (Ce_2C_3);$
- $e 60Fe-10Ce-30C (#4), \times 1000, (\gamma Fe) + (\alpha CeC_2) + (C).$

Fig. 3. X-ray diffraction patterns of annealed alloys in the Fe-Ce-C system:

 $a - Ce_4Fe_4C_7$  (#8),  $\tau_1 + (\gamma Fe) + (\alpha CeC_2)$ ;

- $b Ce_3Fe_5C_6 \ (\#12), \ \tau_1 + (\gamma Fe) + (\alpha CeC_2);$
- $c Ce_{11}Fe_{12}C_{18}$  (#13),  $\tau_1 + (\gamma Fe) + (Ce_2C_3)$ ;
- d 60Fe-10Ce-30C (#4), ( $\gamma$ Fe) + ( $\alpha$ CeC<sub>2</sub>) + (C).

Phase equilibria in the Fe-Ce-C system at 1100°C were studied.

An isothermal section at this temperature was constructed.

The ternary compound  $\tau_1$  is stable at this temperature.

Compositionn of  $\tau_1$  determined as 23Fe-29Ce-48C according our data

|    | Alloy | 7, at.% |      | Heat treatment  | Dhose identification  | Microprobe results, at.% <sup>1</sup> |               |               |
|----|-------|---------|------|-----------------|---|---------------------------------------|---------------|---------------|
| #  | Fe    | Ce      | С    | Heat-treatment  | Phase identification Phase  | Phase                                 | Fe            | Ce            |
| 1  | 87    | 2       | 11   | 1100°C, 35 h    | $\tau_1 + (\alpha CeC_2)$   | -                                     | -             | -             |
| 2  | 85    | 4       | 11   | 1100°C, 85 h    | $\tau_1 + (\alpha CeC_2) + (\gamma Fe)$   | -                                     | -             | -             |
| 3  | 80    | 5       | 15   | 1100°C, 85 h    | $\tau_1 + (\alpha CeC_2)$   | -                                     | -             | -             |
| 4  | 60    | 10      | 30   | 1100°C 24 h     | $(\alpha F_{0}) \pm (\alpha C_{0}C_{0}) + (C)$  | (γFe) 99.4±0                          | 99.4±0.1      | 0.6±0.1       |
| 4  | 00    | 10      | 30   | 1100 C, 24 II   | $(\gamma Fe) + (uCeC_2) + (C)$  | (C)                                   | $0.0 \pm 0.0$ | $0.0\pm0.0$   |
| 5  | 45    | 15      | 40   | 1100°C 24 h     | $(\gamma Fe) + (\alpha CeC_2) + (C)$  | (yFe)                                 | 99.3±0.1      | 0.7±0.1       |
| 5  | 43    | 15      | 40   | 1100  C, 24  II |   | (C)                                   | $0.0\pm0.0$   | $0.0\pm0.0$   |
| 6  | 60    | 16      | 24   | 1100°C, 35 h    | $(\gamma Fe) + (Ce_2C_3)$   | -                                     | -             | -             |
| 7  | 15    | 25      | 60   | 1100°C, 24 h    | $(\gamma Fe) + (\alpha CeC_2) + (C)$  | -                                     | -             | -             |
| 8  | 26.7  | 26.7    | 46.6 | 1100°C, 24 h    | $\tau_1 + (\alpha CeC_2) + (\gamma Fe)$   | $	au_1$                               | 44.1±0.1      | 55.9±0.1      |
|    |       |         |      |                 |   | (yFe)                                 | 98.5±0.1      | $1.5 \pm 0.1$ |
| 0  | 28.6  | 28.6    | 128  | 1100°C 24 h     | $\tau + (C_2, C_1) + (wE_2)$  | $	au_1$                               | 43.8±0.3      | 56.2±0.3      |
| 9  | 20.0  | 28.0    | 42.0 | 1100 C, 24 II   | $t_1 + (Ce_2C_3) + (\gamma re)$   | (yFe)                                 | 98.6±0.1      | $1.4{\pm}0.1$ |
| 10 | 18.2  | 27.3    | 54.5 | 1100°C, 35 h    | $(\alpha CeC_2) + (\gamma Fe)$  | $(\alpha CeC_2)$                      | 0.5±0.3       | 99.5±0.3      |
| 11 | 25    | 25      | 50   | 1100°C, 35 h    | $(\alpha CeC_2) + (\gamma Fe)$  | -                                     | -             | -             |
| 12 | 357   | 21.4    | 12.0 | 1100°C 35 h     | $\frac{(\alpha \text{CeC}_2) + (\gamma \text{Fe})}{\tau_1 + (\alpha \text{CeC}_2) + (\gamma \text{Fe})} \frac{\tau_1}{(\alpha \text{CeC}_2)}$ | 44.1                                  | 55.9          |               |
| 12 | 55.7  | 21.4    | 42.9 | 1100 C, 55 II   |   | $(\alpha CeC_2)$                      | 1.1           | 98.9          |
| 13 | 29.3  | 26.8    | 43.9 | 1100°C, 35 h    | $\tau_1 + (Ce_2C_3) + (\gamma Fe)$  | $	au_1$                               | 44.0±0.1      | 56.0±0.1      |
| 14 | 16.7  | 31.2    | 52.1 | 1100°C, 35 h    | $\tau_1 + (\alpha CeC_2) + (Ce_2C_3)$   | $	au_1$                               | 43.8±0.1      | 56.2±0.0      |
| 14 |       |         |      |                 |   | $(Ce_2C_3)$                           | 0.5±0.1       | 99.5±0.1      |
| 15 | 10    | 33      | 57   | 1100°C, 35 h    | $\tau_1 + (\alpha CeC_2) + (Ce_2C_3)$   | $	au_1$                               | 44.0±0.0      | 56.0±0.1      |
| 15 |       |         |      |                 |   | $(Ce_2C_3)$                           | 0.4±0.1       | 99.6±0.1      |
| 16 | 9.4   | 34.4    | 56.2 | 1100°C, 35 h    | $\tau_1 + (\alpha CeC_2) + (Ce_2C_3)$   | -                                     | -             | -             |

Table 1. Phase identification and composition of the Fe-Ce-C phases according to the EPMA examination of annealed at 1100°C for 24-85 h alloys

<sup>1</sup> The ratio of metal contents Fe/Ce was determined for the phases, since the error of the carbon content determination exceeds the error of the metal determination

| Phase                               | Crystal structure  | Lattice parameters, Å                | Alloy   | Refs.  |
|-------------------------------------|--|--------------------------------------|---|--------|
| δFe                                 | W,<br>A2, cI2-Im3m   | a = 2.9315                           | -   | [37]   |
| γFe                                 | Cu,<br>A1, cF4-Fm-3m   | a = 3.6467                           | at 915°C  | [37]   |
| αFe                                 | W,   | <i>a</i> = 2.8665                    | at 25°C   | [37]   |
|                                     | A2, cI2-Im3m   | a = 2.863(2)                         | 60Fe-30Ce-10C, 1100°C / 24 h  | Th. w. |
|                                     |  | a = 2.866(1)                         | Ce <sub>4</sub> Fe <sub>4</sub> C <sub>7</sub> (#8), 1100°C / 24 h            | Th. w. |
|                                     |  | a = 2.865(2)                         | Ce <sub>2</sub> Fe <sub>2</sub> C <sub>3</sub> (#9), 1100°C / 24 h            | Th. w. |
|                                     |  | a = 2.867(1)                         | $Ce_{3}Fe_{5}C_{6}$ (#12), 110°C / 35 h                                       | Th. w. |
| εFe                                 | Mg,<br>A3, <i>hP2-P6</i> <sub>3</sub> / <i>mmc</i>                                       | a = 4.68, c = 3.96                   | at 25°C, 13 GPa   | [37]   |
| δCe                                 | W,<br>A2, cI2-Im3m   | <i>a</i> = 4.12                      | -   | [37]   |
| γCe                                 | Cu,<br>A1, cF4-Fm-3m   | <i>a</i> = 5.1610                    | -   | [37]   |
| βCe                                 | αLa,<br>A3', <i>hP</i> 4- <i>P</i> 6 <sub>3</sub> / <i>mmc</i>                           | <i>a</i> = 3.6810, <i>c</i> = 11.857 | -   | [37]   |
| αCe                                 | Cu,<br>A1, cF4-Fm-3m   | <i>a</i> = 4.85                      | -   | [37]   |
| (C)                                 | C-graphite,<br>A9, hP4-P6 <sub>3</sub> /mmc  | a = 2.4612, c = 6.7090               | -   | [37]   |
| Fe <sub>17</sub> Ce <sub>2</sub> ht | $Zn_{17}Th_2$ ,  | a = 8.485, c = 12.433                | -   | [38]   |
|                                     | hR57-R-3m  | <i>a</i> = 8.496, <i>c</i> = 12.414  | -   | [3]    |
| $Fe_{17}Ce_2C_x$                    | $Pr_2Mn_{17}C_{1.77}$ ,  | a = 8.72, c = 12.64                  | 1170 K / week   | [39]   |
|                                     | hR66-R-3m  | a = 8.73, c = 12.56                  | -   | [40]   |
|                                     |  | a = 8.540, c = 12.424                | 1100°C / several weeks  | [41]   |
|                                     |  | a = 8.534, c = 12.436                | 1170 K / 20-30 days   | [42]   |
|                                     |  | a = 8.74, c = 12.65                  | x=2.8   | [43]   |
| Fe <sub>17</sub> Ce <sub>2</sub> rt | Th <sub>2</sub> N1 <sub>17</sub> ,<br><i>hP</i> 38- <i>P</i> 6 <sub>3</sub> / <i>mmc</i> | a = 8.49, c = 8.281                  | -   | [13]   |
| Fe <sub>2</sub> Ce                  | MgCu <sub>2</sub> ,  | <i>a</i> = 7.296                     | -   | [38]   |
|                                     | <i>C</i> 15, <i>cF</i> 24- <i>F</i> d-3 <i>m</i>   | <i>a</i> = 7.302                     | -   | [44]   |
| $Ce_2C_3$                           | $Pu_2C_3$ ,  | a = 8.4480                           | Ce-rich   | [15]   |
|                                     | $D5_{c}, cI40-I-43d$   | a = 8.450                            | C-rich  | [15]   |
|                                     |  | a = 8.446(1)                         | $Ce_2Fe_2C_3C$ (#9), 1100°C / 24 h  | Th. w. |
| $pCeC_2$                            | $CaF_2$ ,<br>C1 = aF12 Fm 2m   | a = 5.939                            | -<br>   | [15]   |
|                                     | C1, CF 12-F m-Sm   | a = 5.927                            | at 1110 C   | [19]   |
| aCeC.                               | CaC  | a = 0.023                            | at 2000 C   | [19]   |
| uccc <sub>2</sub>                   | C11, $tI6$ -L/mmm  | $a = 3.875 \ c = 6.489$              | at 580°C in equil with $Ce_2C_2$  | [15]   |
|                                     |  | a = 3.878(1) $c = 6.489(2)$          | $60\text{Fe}-30\text{Ce}-10\text{C}$ (#4) $1100^{\circ}\text{C}/24 \text{ h}$ | Th w   |
| CeC <sub>6</sub>                    | hP14(?)-P6 <sub>3</sub> /mmc   | a = 4.490, c = 13.9526               | -   | [17]   |
| $Ce_4Fe_4C_7$                       | <i>tP</i> *  | a = 7.22, c = 5.82                   | Ce <sub>4</sub> Fe <sub>4</sub> C <sub>7</sub> , 800°C / 300 h                | [8]    |
| $(Ce_3Fe_5C_6)$                     | $Nd_3Fe_5C_6$ ,  | a = 7.231, c = 3.192                 | Ce <sub>3</sub> Fe <sub>5</sub> C <sub>6</sub> , 1050°C                       | [21]   |
|                                     | P-4m2  | a = 7.232(2), c = 3.192(1)           | Ce <sub>4</sub> Fe <sub>4</sub> C <sub>7</sub> (#8), 1100°C / 24 h            | Th. w. |
|                                     |  | a = 7.231(2), c = 3.191(2)           | Ce <sub>2</sub> Fe <sub>2</sub> C <sub>3</sub> (#9), 1100°C / 24 h            | Th. w. |
|                                     |  | a = 7.235(3), c = 3.194(2)           | Ce <sub>3</sub> Fe <sub>5</sub> C <sub>6</sub> (#12), 1100°C / 35 h           | Th. w. |
| Ce <sub>3.67</sub> FeC <sub>6</sub> | $La_{3.67}FeC_6$ ,   | a = 8.686, c = 5.309                 | 800-1000°C / 5-9 weeks  | [37]   |
| $(Ce_2Fe_2C_3)$                     | <i>hP</i> 24- <i>P</i> 6 <sub>3</sub> / <i>m</i>   | a = 8.7926(8),<br>c = 16.0459(15)    | for La <sub>3.67</sub> FeC <sub>6</sub>                                       | [38]   |
| Ce <sub>2</sub> Fe <sub>14</sub> C  | $Nd_{2}Fe_{14}B,$<br>tP68-P4 <sub>2</sub> /mnm   | a = 8.74, c = 11.85                  | annealed at 970 K / 0.5 h   | [45]   |

Table 2. The crystal structures and lattice parameters of the Fe-Ce-C phases

<sup>1</sup> Th. w. - results of this work







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