ISSN 2409-9066. Sci. innov. 2016, 12(1): 22—27 doi: http://dx.doi.org/10.15407/scine12.01.022

Ponomarenko, O.M., Brik, O.B., and Dudchenko, N.O.
Semenenko Institute of Geochemistry, Mineralogy and Ore Formation, the NAS of Ukraine, Kyiv

FACILITY FOR TRANSFORMATION OF MAGNETIC PROPERTIES OF WEAK MAGNETIC OXIDIZED IRON ORES IN ORDER TO IMPROVE TECHNOLOGIES FOR IRON ORE CONCENTRATE PRODUCTION

New facility for continuous recording of iron ore magnetization intensity depending on temperature, when heating iron ores in the presence of reducing agent has been designed. The facility enables recording the transformation of weak magnetic minerals into strong magnetic ones under the influence of reducing agents and temperature, as well as determining the Curie temperature of the minerals. Using the facility it has been showed that heating of goethite and hematite in the presence of 4% starch within the temperature range of 300–600 °C leads to a significant increase in the sample magnetization intensity. X-ray diffraction has confirmed that under the mentioned conditions the structure of hematite and goethite transforms into the magnetite structure. Obtained results open up new possibilities for the development of effective technologies for oxidized iron ore beneficiation.

Keywords: magnetite, hematite, goethite, and structure transformation.

Ukraine is the seventh among the world countries by iron ore deposits consisting of mainly magnetite and hematite quartzites. The magnetite quartzite easily takes beneficiation by magnetic separation method and is widely used for production of iron ore concentrates. As of today, the magnetite ore deposits has been substantially exhausted. The poor hematite quartzite extracted together with the magnetite quartzite often are unsuitable for beneficiation by the magnetic separation method as well as by other methods. The tailings are piled and occupy large areas, which cause environment, economic, and, in the future, social problems. According to estimates [1], the explored deposits of oxidized ferruginous quartzite in the Kryvyi Rih Iron Ore Field total 5 billion tons. However, even the material kept in dumps and tailing facilities (240 million ton ore) is enough for many years. None country has such deposits of raw materials ready for processing.

In the industry, for beneficiating the oxidized iron ores a technique based on the use of reducing agents, namely, mix of carbon monoxide and hydrogen at a high temperature (900–1450 °C) is used [2]. This technique enables to get magnetic material from nonmagnetic oxidized iron ores. The former is further used for obtaining iron-containing concentrate by the magnetic separation method. However, this method is very power intensive therefore, it is necessary to design new energy efficient techniques for processing the poor magnetic ferrous materials on which new methods for oxidized iron ore beneficiation will be based. In order to address this problem it is necessary to design laboratory facilities for studying the processes related to conversion of poor magnetic ferrous oxides and hydroxides into the strong magnetic iron oxides (magnetite) under the action of various external factors. This research is aimed at creating laboratory facilities for continuous recording of magnetic material and ore magnetization intensity depending on temperature, during their chemical transformation under conditions of reduction.

RESEARCH MATERIALS AND METHODS

The basic experiments were carried out for hematite and goethite ore samples taken from the Kryvyi Rih deposits. The samples were milled to the fractions less than 0.1 mm. For studying the phase transitions, the samples of goethite and hematite ores were carefully mixed with 4% starch, placed into a quartz mini-reactor and heated up to 650 °C /cooled at a rate of 65–80 °C/min.

The sample structural transformations were studied by the X-ray diffraction method (DRON-3M XRD meter with monochromator) with copper anticathode (CuK_α, λ = 1.54178 Å).

DISCUSSION OF RESULTS

The thermomagnetic research was carried out at a facility designed for the study of transformations of poor magnetic ferrous oxides and hydroxides into the strong magnetic oxides and the identification of magnetic phases of minerals [4] (Fig. 1). The device records the sample magnetization intensity depending on temperature, during its chemical transformation, under conditions of reduction, and automatically identifies the Curie temperature that is ferromagnetic constant. The Curie temperature unambiguously depending on the mineral crystalline structure, its value can be used to identify magnetic minerals in the ores and magnetic materials.

Device components:

1. Magnetic digital balance with a weight limit of 210 g, readability of 0.001 g, and digital output for communication with PC. The device is used for measuring the interaction between the sample and the magnetic field.

2. Digital thermo-controller (DTC) for measuring and controlling temperature (*DTC 02 Universal+*). The device can be connected to PC, work with network, and record process parameters.

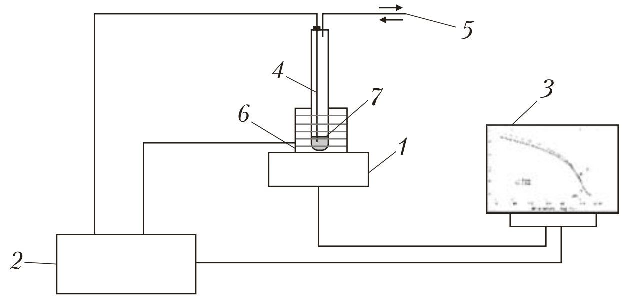

Fig. 1. Diagram of device for study of the magnetic phases of magnetically ordered minerals and their identification. The explanations are given in the text

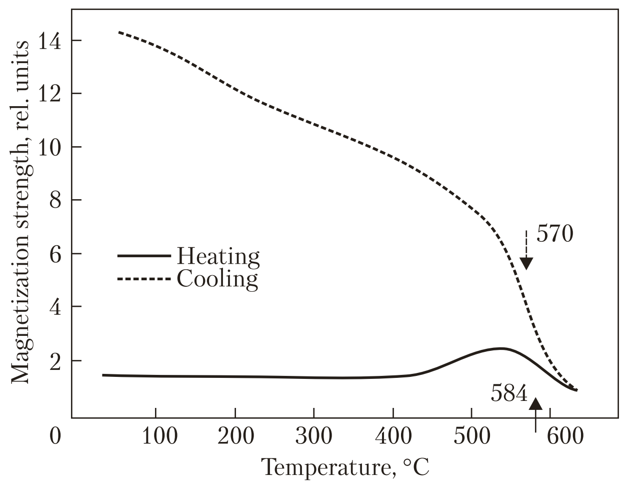

Fig. 2. Thermomagnetic curve for hematite ore with 4% starch

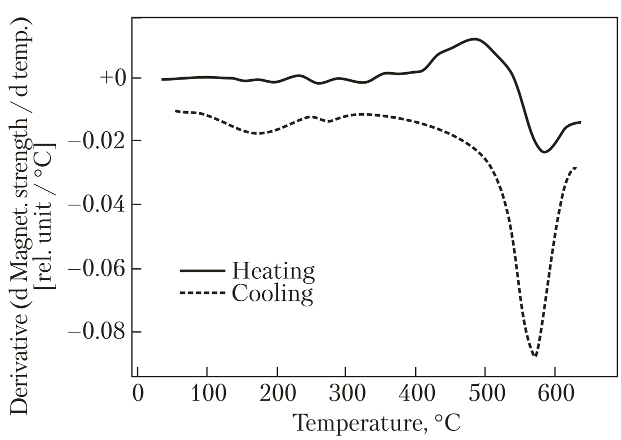

Fig. 3. Differential thermomagnetic curve for hematite ore with 4% starch

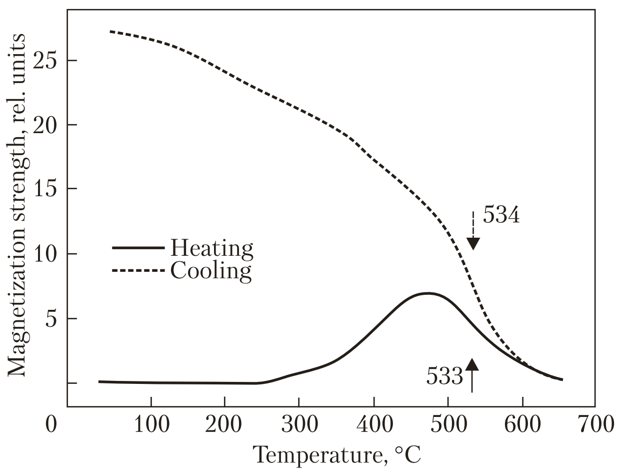

Fig. 4. Thermomagnetic curve for goethite ore with 4% starch

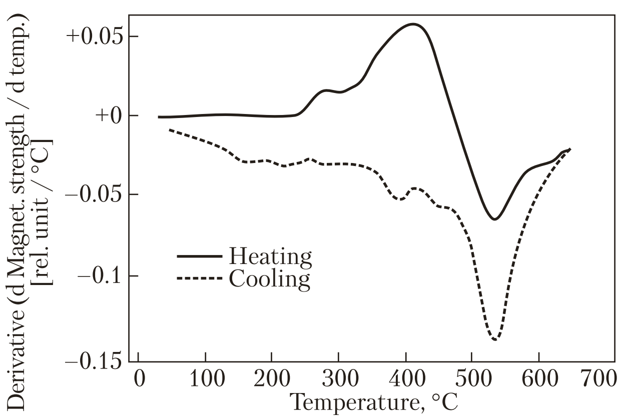

Fig. 5. Differential thermomagnetic curve for goethite ore with 4% starch

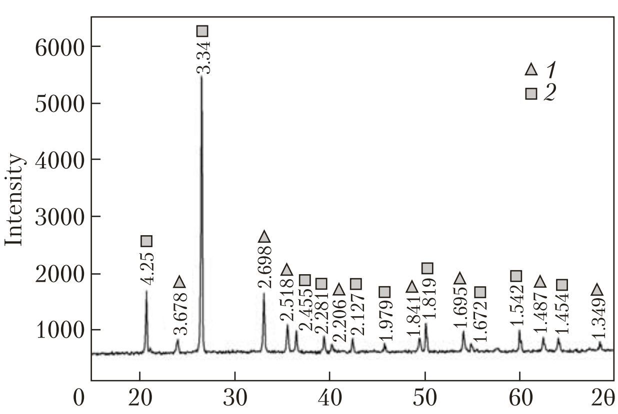

Fig. 6. XRD pattern of the initial hematite quartzite sample (*1* — hematite Fe_2O_3; *2* — quartz)

3. PC for transferring and collecting data on time, temperature (set by DTC and measured by thermocouple), and recording data from magnetic digital balance. The data are processed with the help of special software storing them on the computer.

4. Thermocouple based on *chromel–copel* alloy, which transfers measured temperature to the thermo-controller.

5. Pipe for removing gaseous substances (flexible rubber pipe having a water trap at the end).

6. Nichrome solenoid as heating element (helix height is 1 cm, wire diameter is 0.8 mm). It is controlled by thermo-controller and operates within the range from ambient temperature to 650 °C.

7. Reactor with the sample (a quartz vessel with rubber plug, duct for gases, and hole for thermocouple). The vessel diameter is 1 cm. The sample is a powder mixture. Having been prepared it is put into the reactor having a height of approximately 1 cm (height of solenoid).

For measuring the sample magnetization intensity with the help of this facility, the device based on the measurement of interaction between the sample and the magnetic field of constant magnet is used. The constant magnet based on NdFeB alloy is fixed on a measuring sensor with standard digital output to PC for recording the interaction between the sample and the magnetic field of constant magnet. Heating and cooling of the sample are programmed by temperature control unit with the help of PC software. The dependence of magnetization intensity on temperature and the Curie temperature are built and determined by PC. The structure transformations take place in the quartz micro-reactor designed in such a way as to ensure supply and removal of gaseous oxidants and reducing agents. The solid oxidants and reducing agents are added in required quantity when the sample is prepared for the study. The Curie temperature is computed on the basis of the extreme points on the curve of magnetization intensity first derivative by temperature.

The dependence of magnetization intensity on temperature for hematite ore and the corresponding curve of the first derivative recorded with the

ISSN 2409-9066. Sci. innov. 2016, 12(1)

help of designed facility are showed in Figs. 2 and 3, while for the goethite ore, they are given in Figs. 4 and 5, respectively.

One can see from Figs. 2, 3, 4 and 5 that the date enable determining the temperature of beginning of chemical transformations of the hematite and goethite weak magnetic phases into the strong magnetic phase, as well as the sample magnetization intensity after cooling down to ambient temperature and the Curie temperature of the obtained strong magnetic phase (arrows in Figs).

New magnetic phase with Curie temperature of 570 and 530 °C has been showed to appear as a result of structural transformation of hematite and goethite ores, respectively. The obtained temperature is close to the Curie temperature of magnetite (580 °C).

The analysis of X-ray diffraction pictures before and after transformations has showed that the structure of weak magnetic minerals (hematite and goethite) transforms into the structure of strong magnetic mineral (magnetite). Reflexes on the diffraction picture of initial hematite ore (Fig. 6, respective interplanar space *d*(Å) are: for hematite 3.678; 2.698; 2.518; 2.206; 1.841; 1.695; 1.487 and for quartz: 4.25; 3.34; 2.455; 2.281; 2.127; 1.979; 1.819; 1.542; 1.454) show that the sample contains hematite and quartz traces. The magnetite reflexes have been showed to appear on the diffraction picture of the sample obtained, after the structural transformation of hematite ore with the help of the designed facility (Fig. 7) (for magnetite, the interplanar spaces *d* (Å) are: 2.964; 2.529; 2.099; 1.616).

The reflexes on the diffraction picture of the goethite sample (Fig. 8) are referred to goethite (respective interplanar spaces for goethite *d* (Å) are: 4.26; 455; 722), hematite (respective interplanar spaces for hematite *d* (Å) are: 2.702; 2.201; 1.697; 1.451), quartz (respective interplanar spaces for *quartz d* (Å) are: 4.181; 3.345; 1.819; 1.543; 1.373), and kaolinite (respective interplanar spaces for kaolinite *d* (Å): 3.576).

After the transformation of goethite ore with the help of the facility designed, on the diffraction

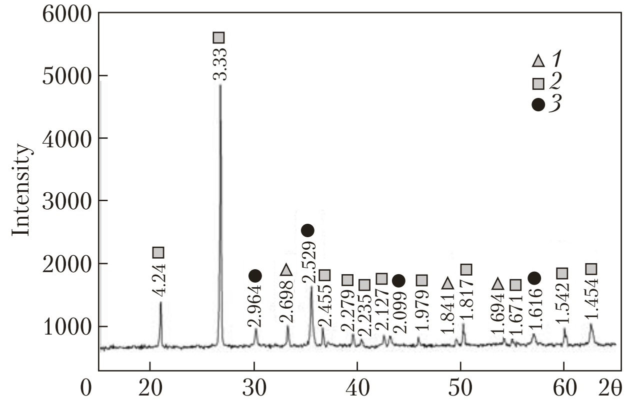

Fig. 7. XRD pattern of the sample after hematite structural transformation (*1* — hematite Fe_2O_3; *2* — quartz; *3* — magnetite Fe_3O_4)

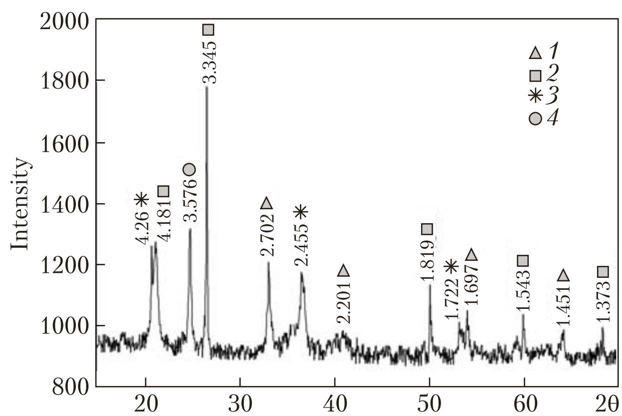

Fig. 8. XRD pattern of initial goethite sample (*1* — hematite Fe_2O_3; *2* — quartz; *3* — goethite γ-FeOOH, *4* — kaolinite)

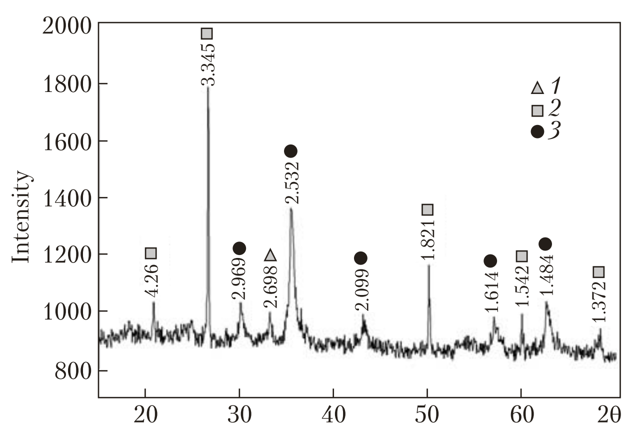

Fig. 9. XRD pattern of the sample after goethite structural transformation (*1* — hematite Fe_2O_3; *2* — quartz; *3* — magnetite Fe_3O_4)

picture of the sample (Fig. 9), the goethite reflexes disappear, whereas the magnetite ones come into being (for magnetite, the interplanar spaces d (Å) are: 2.969; 2.532; 2.099; 1.614; 1.484).

Hence, the study of iron ores on the designed facility has showed that as a result of heating of hematite and goethite ores in the presence of 4% starch, within the range of temperature 300–600 °C, hematite and goethite transform into magnetite.

CONCLUSIONS

1. A laboratory facility for recording the magnetization intensity depending on temperature (up to 650 °C) in the presence of reducing agents and determining the Curie temperature of samples has been designed.

2. With the help of the above facility it has been showed that under heating (up to 650 °C) hematite and goethite ores in the presence of starch as reducing agent change their structure and magnetic properties.

3. The new facility for continuous record of magnetization intensity of iron ores depending on temperature when heating in the presence of reducing agent can be recommended for developing new techniques of beneficiation of oxidized iron ores from the Kryvyi Rih deposits and for managing manmade iron ore dumps and tailings.

The research was done within the framework of R&D project of the NAS of Ukraine no.10 in 2014.

REFERENCES

1. Byzov V.F. Razrabotka tehnologii kombinirovannogo obogashhenija okislennyh rud. *Gornaja promyshlennost'*. 2007, N 4: 30—34 [in Russian].
2. *Pat. US № 3954444, МПК C21B 5/06.* Process for the direct reduction of iron ores. W. Wenzel, P. Speih, F.H. Franke, B. Dreyer, 1976.
3. *Pat. Ukrai'ny № 28424, MPK G01G 49/08.* Sposib otrymannja magnetytu. Polishhuk V.Ju., Dem'janchuk B.O., Polishhuk I.V. [in Ukrainian].
4. *Pat. Ukrai'ny na korysnu model' № 94514.* Prystrij dlja vyznachennja temperatury Kjuri ta identyfikacii' magnitnyh mineraliv v rudah ta magnitnyh materialah. Janyshpol's'kyj V.V., Aljeksejcev Ju.O., Dudchenko N.O., Virko S.V., Ponomarenko O.M., Bryk O.B. [in Ukrainian].

О.М. Пономаренко, О.Б. Брик, Н.О. Дудченко

Інститут геохімії, мінералогії та рудоутворення ім. М.П. Семененка НАН України, Київ

СТВОРЕННЯ УСТАНОВКИ ДЛЯ ПЕРЕТВОРЕННЯ МАГНІТНИХ ХАРАКТЕРИСТИК СЛАБОМАГНІТНИХ ОКИСНЕНИХ ЗАЛІЗНИХ РУД, В ЗВ ЯЗКУ З УДОСКОНАЛЕННЯМ ТЕХНОЛОГІЙ ВИРОБНИЦТВА ЗАЛІЗОРУДНИХ КОНЦЕНТРАТІВ

Створено нову установку для неперервної реєстрації намагніченості залізних руд в залежності від температури при їх нагріванні у відновлювальних умовах. Установка дозволяє реєструвати процеси перетворень слабомагнітних мінералів в сильномагнітні під впливом відновників та температури, а також визначати температуру Кюрі мінералів. За допомогою створеної установки показано, що нагрівання гетиту та гематиту в присутності 4% крохмалю в діапазоні температур 300–600 °С веде до суттєвого збільшення намагніченості досліджених зразків. Методом дифракції рентгенівських променів підтверджено, що за зазначених умов структура гематиту та гетиту перетворюється на структуру магнетиту. Отримані результати надають нових можливостей для розробки ефективних технологій збагачення окислених залізних руд.

Ключові слова: магнетит, гематит, гетит, перетворення структури.

А.Н. Пономаренко, А.Б. Брик, Н.А. Дудченко

Институт геохимии, минералогии и рудообразования им. Н.П. Семененко НАН Украины, Киев

СОЗДАНИЕ УСТАНОВКИ ДЛЯ ПРЕОБРАЗОВАНИЯ МАГНИТНЫХ ХАРАКТЕРИСТИК СЛАБОМАГНИТНЫХ ОКИСЛЕННЫХ ЖЕЛЕЗНЫХ РУД, В СВЯЗИ С УСОВЕРШЕНСТВОВАНИЕМ ТЕХНОЛОГИЙ ПРОИЗВОДСТВА ЖЕЛЕЗОРУДНЫХ КОНЦЕНТРАТОВ

Создана новая установка для непрерывной регистрации намагниченности железных руд в зависимости от температуры при их нагревании в восстановительных условиях. Установка позволяет регистрировать процессы преобразования слабомагнитных минералов в сильномагнитные под влиянием восстановителей и температуры, а также определять температуру Кюри минералов. С помощью созданной установки показано, что нагревание гетита и гематита в присутствии 4% крахмала в диапазоне температур 300—650 °C приводит к существенному увеличению намагниченности исследуемых образцов. Методом дифракции рентгеновских лучей подтверждено, что при указанных условиях структура гематита и гетита преобразовывается в структуру магнетита. Полученные результаты открывают новые возможности для разработки эффективных технологий обогащения окисленных железных руд.

Ключевые слова: магнетит, гематит, гетит, преобразования структуры.

Received 22.06.15