MATERIALS FOR CARBON CAPTURE AND STORAGE TECHNOLOGIES

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Abstract

Carbon dioxide produced by human activities is one of the major contributors to the greenhouse effect. A way to reduce air pollution could be CCS - Carbon Capture and Storage Technology. Some of the CCS technologies require high operating temperatures (up to 900°C) that mean higher demands on corrosion resistance and other properties of selected construction materials. The main target of this study is to describe an experimental device simulating the CCS environment for construction materials testing and the study of high-temperature corrosion processes in gaseous environments.

Key words: *CCS technology, corrosion, heat resistant materials*

1. INTRODUCTION

Industrial processes are often associated with high production of carbon dioxide, which is a well-known greenhouse gas. The high concentration of CO_2 in the atmosphere has negative effects on the environment, therefore it is necessary to reduce its emissions. Carbon capture and storage technologies could be a perspective solution to reduce CO_2 emissions and positively influence global climate. CCS technologies consist of the separation of CO_2 from industrial and energy-related sources, transport to a storage location and long-term isolation from the atmosphere. (Figure 1.)

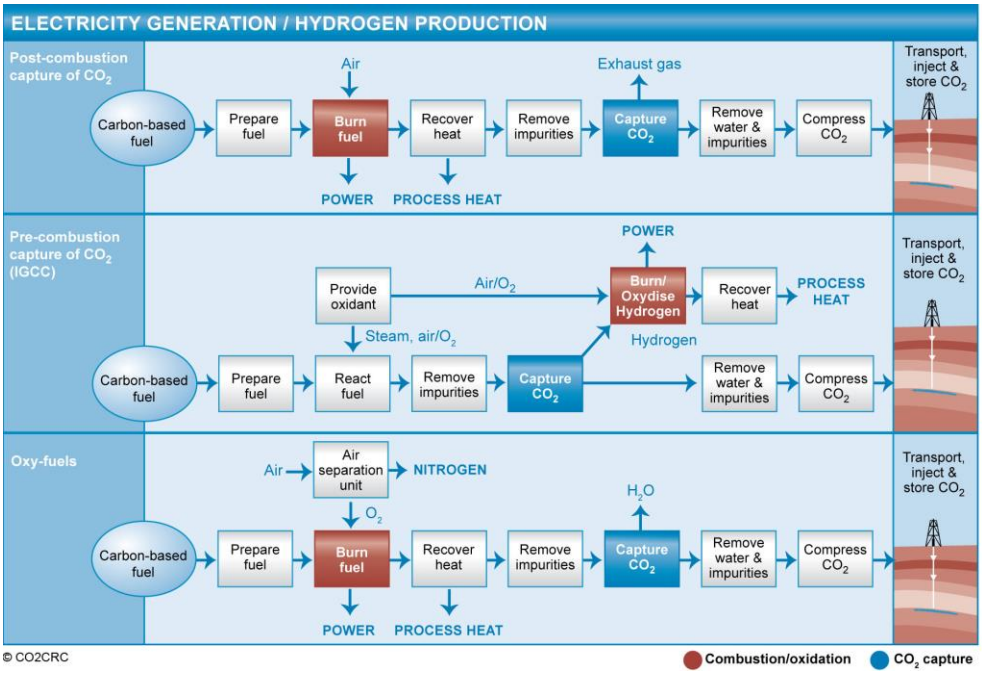

Figure 1. Pre-combustion and post-combustion processes for carbon dioxide capture (CO2CRC)

There are different types of CO_2 capture systems: post-combustion, pre-combustion and oxyfuel combustion. Afterwards the CO_2 should be compressed and transported for storage in geological formations, in the ocean, or for use in industrial processes. (IPCC 2005)

As the most economical method seems to be the technology of CO_2 capture from flue gas using a sorbent - mostly CaO. The mechanism of capture and release takes place in two steps (Huang et al. 2011):

1) Carbonation: CO_2 from the flue gas reacts with the sorbent CaO, the temperature in the device is from 450 to 750°C

$$CaO + CO_2 \rightarrow CaCO_3 \quad (1)$$

2) Calcination of limestone takes place at temperatures from 850 to 1000°C

$$CaCO_3 \rightarrow CaO + CO_2 \quad (2)$$

The utilization of CCS technologies will depend on the level of technological development, environmental impact, economic factors and other social influences. This article shows how to increase the corrosion resistance level of technological equipment and extend the life of CCS devices using materials resistant to high temperatures. An experimental device with regard to conditions in the calciner was proposed for the testing of construction materials.

The presented work about materials research for CCS technology is the part of the project "Research of high temperature CO_2 sorption from flue gas using carbonate loop" (acronym HITECARLO, supported by Norway grants). Hitecarlo is the research project with participation of three organizations with technical and engineering focus: University of Chemistry and Technology Prague, Czech Technical University in Prague and The Nuclear Research Institute Řež, a.s.

The main aim of the project is to contribute to the research on application of Carbon capture and storage (CCS) in the Czech Republic. In accordance with the requirements of the European Commission and the SET-Plan (European Strategic Energy Technology Plan) the specific aim of the project is focused on the research and the verification of CO_2 capture technologies using high-temperature carbonate loops in laboratory conditions and the preparation of pilot plant design documentation for its construction on this principle. At the same time, the project contributes to the creation of a knowledge- and personnel-base for CCS processes in the Czech Republic. (University of Chemistry and Technology Prague 2015)

2. EXPERIMENTAL DEVICE

2.1. Device description

Some of the CCS technologies consider high operating temperatures, which means increasing demands on corrosion resistance and other properties of selected construction materials. Two types of materials can correspond with these requirements: heat resistant stainless steels and nickel superalloys. A high-temperature device was built for the tests of the chosen materials. It consists of three basic parts: the first part is a gas bottle with experimental gas mixture and water tank for moistening the gas. The second part of the device includes gas analysers (an oximeter and a humidity meter are included for basic experiments; additional analysers can be also included in case of complicated experimental gaseous environment), furthermore flow meters, valves and other components. Finally, the most important part of the device is a high temperature tube furnace with a quartz retort (internal diameter 80 mm). The devices of this type can be used especially for experiments at high temperatures up to 1000°C and tests in gaseous environment with the possibility of water injection.

The individual parts of the device and their arrangement can be seen in Figure 2.

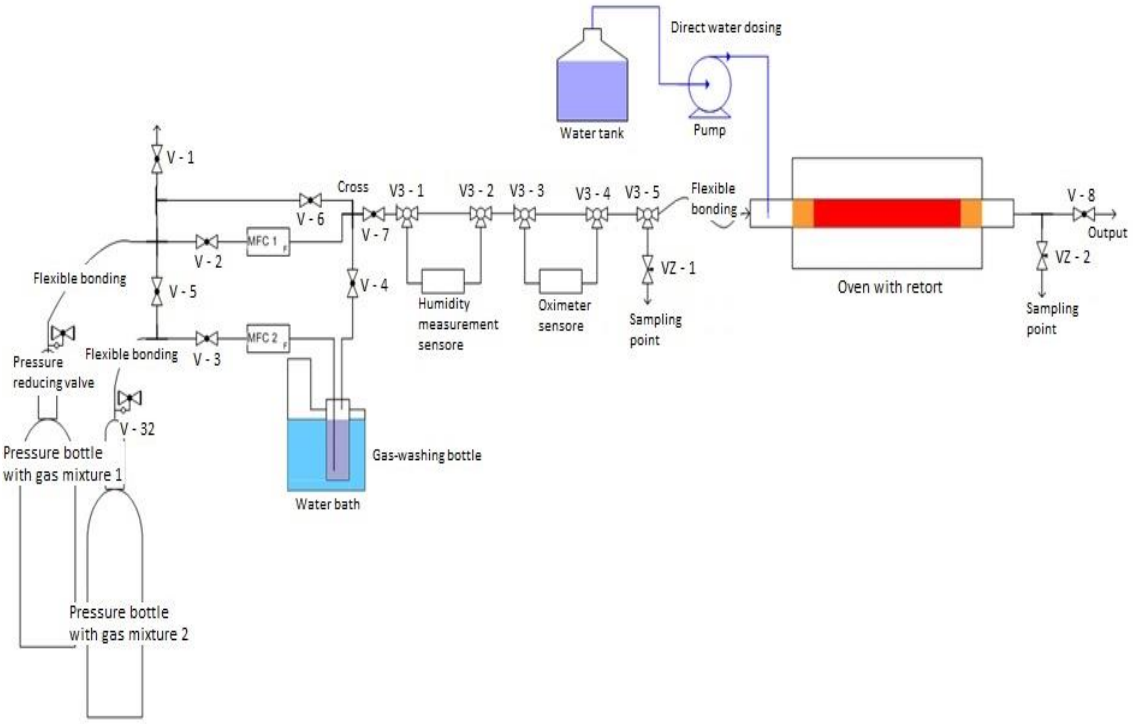

Figure 2. Schema of experimental device

2.2. Tested materials

Two types of materials were selected for high temperature corrosion tests in gaseous environments of CCS technologies: heat resistant stainless steels and nickel based alloys. The materials were chosen in accordance to their properties and availability. There were selected AISI 304, AISI 316L, 316Ti and P91 as fireproof stainless steel, which are corrosion-resistant to high temperature and they are easily machinable. As the nickel based alloys were selected Inconel 713, Inconel 738 and Incoloy 800H, which are high strength, resistant to carburizing – they have stability at high temperature, but their disadvantage is difficult machinability. From the materials were made corrosion coupons with the sample size 10x20x3 mm, the coupons were polished and degreased. Sample holders with the coupons were placed into the retort furnace of the experimental device. The chemical composition of materials is shown in Table 1.

The danger for the life-time of the construction material would be not just the high-temperature inside the system equipment, but also the presence of dust, oxygen, CO_2 and other substances obtained in the gaseous medium. Forming and growing of oxide layers can be initiated on material surface in the environment of CCS technologies due to high-temperature corrosion; the change of mechanical properties of material because of decarburising/carburising of surface layers; or damage of material in terms of shut-down in relation to effect of acid environment (CO_2 with humidity).

2.3. Experimental parameters

The samples of materials will be exposed in the furnace under high temperatures and atmospheric pressure in various gaseous environments, that can be present in a calciner. Basic experiments will be realized in environment of pure CO_2, the following experiments will be carried out in environment of CO_2 with contaminants - like sulphur dioxide, oxygen and water. The examples of main experimental environments composition are shown in Table 2.

All experiments will be performed in dry gas as well as in gas with a certain moisture content. Exposure time will be approximately 500 hours, the temperatures of exposures will be 600°C and 900°C

Tested materials should be analysed after the exposure and their life-time in CCS environment compared.

Table 1. Chemical composition of tested materials (in weight %) (INOX 2016)

Material/%	Fe	Cr	Ni	C	Mo	Ti	Al	Mn	Si	Co	W	Ta	S	P
AISI 304	base	17-19,5	8-10,5	< 0,07										
AISI 316Ti	base	16,5-18,5	10,5-13,5	< 0,08	2-2,5	0,4-0,7								
AISI 316L	base	16,5-18,5	10-12	<0,03	2-2,5			< 2	< 1				<0,015	< 0,045
P91 (1.4903, AISI A213)	base	8-9,5	< 0,4	0,08-0,12	0,85-1,05		< 0,04	0,3-0,6	<0,5					
Inconel 738	<0,35	15,7-16,3	base	0,15-0,2	1,5-2	3,2-3,7	3,2-3,7			8-9	2,4-2,8	1,5-2		
Inconel 713	<5	11-14	base	0,02	3,5-5,5	1	5,5-6,5	<1						
Incoloy 800H	>39,5	19-23	30-35	0,05-0,1		0,15-0,6	0,15-0,6							

Table 2. Composition of the experimental gaseous environments

No.	Chemical composition /vol. %
1	CO_2 - 99,5%
2	O_2 - 10%, N_2 - 8%, SO_2 - 2%, CO_2 - base
3	O_2 - 6,1%, CO_2 - 13,6%, SO_2 - 0,1%, N_2 - base
4	O_2 - 6,1%, CO_2 - 13,6%, N_2 - base

3. ANALYTICAL METHODS

Main analysing methods would be the specification surface layers composition on exposed samples by XPS, GD-OES and structure analysis by SEM microscopy and metallography.

3.1. X-ray photoelectron spectroscopy (XPS)

The top layer of corrosion products created on tested materials will be identified by XPS method. Samples will be analysed by the XPS with the ESCA Probe P apparatus (Omicron Nanotechnology

Ltd.) equipped with Al Kα (1486.6 eV) X-ray source with monochromator and a hemispherical analyser. The size of the analysed area is approximately 1 mm^2. Ar ions of 5 keV energy are used for ion sputtering. The XPS method is based on the principle of measuring the energy distribution of electrons emitted from surface due to irradiation by primary photons or electrons. The basic components are a source of primary radiation and energy analyser.

Spectra evaluation is carried out by using CasaXPS software. The concentrations of the elements are given in at %.

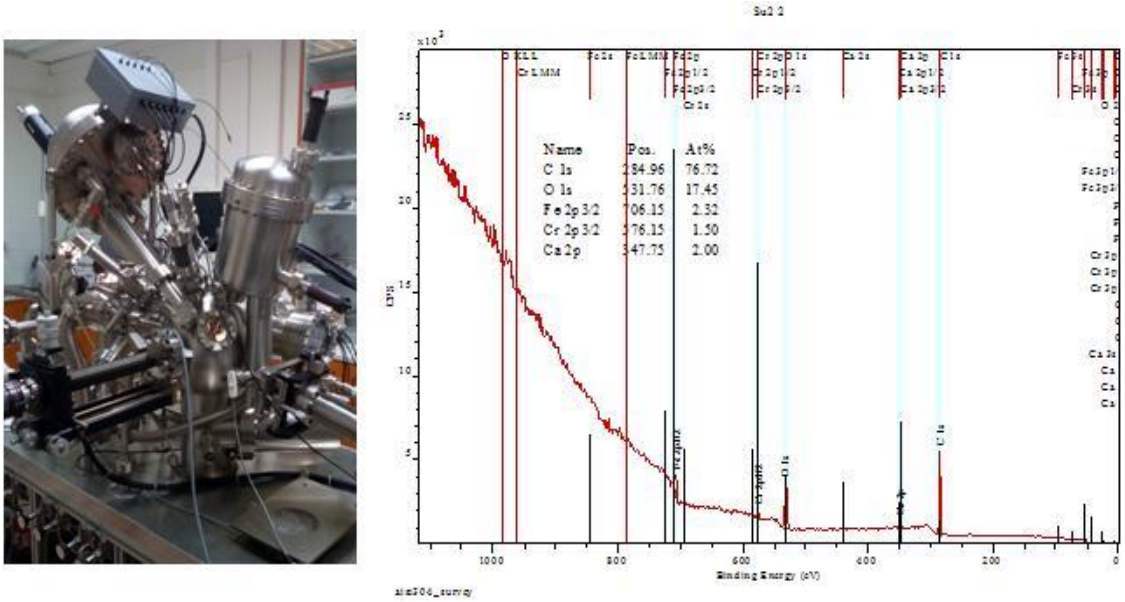

Figure 3. ESCA Probe P apparatus and XPS spectrum from AISI304 (with surface contamination)

3.2. Scanning electron microscopy (SEM)

Microstructures before and after the oxidation tests will be studied using a scanning electron microscope TESCAN VEGA 3 LMU with electron source tungsten filament (cathode) and energy range 30kV. Images of the samples are obtained by scanning the sample with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that contain information about the sample's surface topography and composition.

Magnification in a SEM can be controlled over a range of about 6 orders of magnitude from about 10 to 500,000 times. Unlike optical and transmission electron microscopes, image magnification in a SEM is not a function of the power of the objective lens. (Kazmiruk 2012)

The most common SEM mode is detection of secondary electrons emitted by atoms excited by an electron beam. The number of secondary electrons that can be detected depends, among other things, on the angle at which the beam meets the surface of a specimen (specimen topography). An image displaying the topography of the surface is created by scanning the sample and collecting the secondary electrons. The types of signals produced by an SEM include secondary electrons (SE), back-scattered electrons (BSE), characteristic X-rays (Figure 4). (Reimer 1998)

3.3. Glow discharge optical emission spectroscopy (GDOES)

The GD Profiler 2 (HORIBA Scientific) will be used for surface and profile analysis. Pulsed RF GD-OES is the technique that can provide both surface, depth profile and bulk composition, with high sensitivity to all elements (including the gaseous elements), for almost all solid materials, including metals, metal alloy coatings, semiconductors, polymer coatings, glass, etc. (Figure 5.). It relies on the controlled sputtering of a representative area of the material of interest and the simultaneous detection

of the sputtered species. It is a technique to measure depth profiles of constituent elements in solid samples by detecting emissions from atoms accommodated in plasma by sputtering. This method offers high depth resolution (a few nanometres) and quite quick measurements.

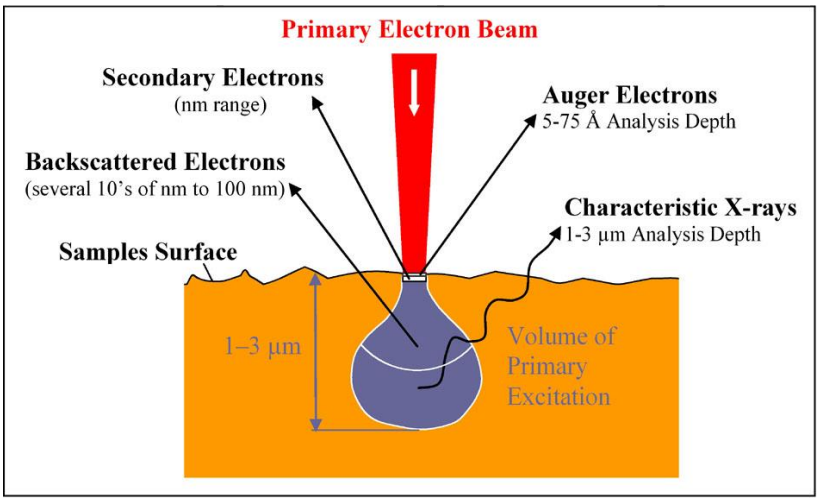

Figure 4. The types of signals produced by an SEM (SE, BSE, X-rays) (Center for Atmospheric Plasma Coatings)

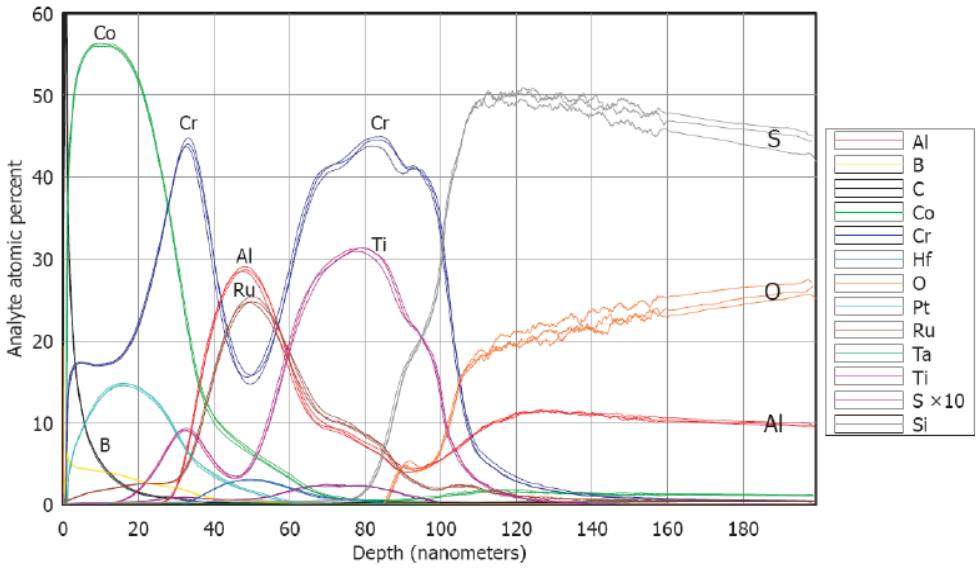

Figure 5. Example of profile analyses (GDOES)

4. CONCLUSION

The main topic of the Hitecarlo project is to upgrade knowledge and technical approaches in CCS technologies. This project is divided to several sections: Experimental research of carbonate looping and appropriate sorbents, Design of pilot facility concept for the research of flue gas decarbonation, Documentation for the construction of a pilot plant for research of flue gas decarbonation, LCA - life cycle assessment and determination of the carbon footprint of high temperature carbonate loop process by the use of LCA, Overview of industrial sources, emitting CO_2 and Investigation of structural materials for the high-temperature carbonate loop technology. (University of Chemistry and Technology Prague 2015)

The material research for CCS technologies is important for the service life of required equipment and to obtain information about the behaviour of construction materials in high temperature and gaseous environment, which will be helpful for another industrial application as well - for example the cooling circuits of 4th generation nuclear reactors.

The high-temperature experimental device was designed for the service life of materials testing. At this moment the optimizing and launch preparation is in progress, as soon as possible the first experiments in high-temperature CO_2 will be performed. The following materials exposures will continue in environments corresponding with the real conditions in CCS technologies.

ACKNOWLEDGEMENT

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