THERMOGRAVIMETRIC ANALYSIS OF THE REDUCTION OF ILMENITE AND NU-LHT-2M WITH HYDROGEN AND METHANE. P. Reiss[1], F. Kerscher[2] and L. Grill[1], [1]Technical University of Munich, Institute of Astronautics, Boltzmannstr. 15, 85748 Garching, Germany (p.reiss@tum.de), [2]Technical University of Munich, Institute for Energy Systems, Boltzmannstr. 15, 85748 Garching, Germany.

Introduction: The majority of past studies on oxygen production from lunar regolith has been focussing on the chemical reduction of the regolith using hydrogen as a reactant gas. Another process that has often been discussed for producing oxygen from lunar regolith is the chemical reduction with methane. In the past, these reduction processes have been studied theoretically and experimentally on different scales, with different sample materials, and different process conditions. Although both processes have different characteristics and requirements, it is not entirely understood to what extent the reactant gases could potentially be replaced with each other in an ISRU reactor and what the implications in terms of oxygen yield, reaction rates, and by-products are depending on the feedstock. For an ISRU reactor it is advantageous to avoid temperatures in the melting range of lunar regolith, above 1100 °C [1]. It is questionable if and how well the reduction with methane works below this temperature. To enable the evaluation and selection of ISRU processes on a common basis we performed a direct comparison of the above processes under the same reaction conditions by means of thermogravimetric analysis (TGA).

Materials and methods: The investigated samples were 93-96 % pure ilmenite ($FeTiO_3$) as a reference with good susceptibility to reduction and the lunar regolith simulant NU-LHT-2M as a chemically analogue for (polar) highland regolith. They were sieved to the 100-125 µm size fraction to make sure that the ilmenite particles in NU-LHT-2M are included in the sample [2]. The apparatus used for thermogravimetric analysis was a Linseis STA PT 1750. Samples with a mass of 70-80 mg were heated in a flow of nitrogen, hydrogen, and methane at 200 ml/min with a heating rate of 6 °C/min from room temperature to 1000 °C.

Results with hydrogen: The ilmenite sample showed a significant weight loss of 12.7 % between 500 °C and 850 °C due to the reduction with hydrogen, slightly larger than the the stoichiometrically expected weight loss of 10.5 %. An ilmenite sample that was baked out under nitrogen beforehand yielded a similar weight loss of 12.6 %. For the NU-LHT-2M sample a weight loss of 0.7 % was measured between 550 °C and 700 °C. Another NU-LHT-2M sample that was baked out under nitrogen beforehand yielded a weight loss of only 0.2 % between 650 °C and 700 °C. This corresponds well with the expected stoichiometric weight loss for an ilmenite content of 1.5 % [2]. The higher weight loss measured for the first NU-LHT-2M sample is caused by additional thermal decomposition products, such as water, carbon monoxide, and carbon dioxide [3]. Potential side reactions with these products complicate the interpretation of the weight change measured via TGA because they are on the same order as the expected weight loss due to reduction of the ilmenite content. Therefore it is recommended to bake out NU-LHT-2M before using it for ISRU preparation studies. An additional observation was that the NU-LHT-2M treated with hydrogen noticeably changed its visual appearance from white or bright grey to dark grey (Figure 1). This most likely is because of a chemical alteration of plagioclase, which makes up ~60 % of the sample

Results with methane: In order to serve as a reactant, methane needs to be decomposed into hydrogen and carbon. This can happen at temperatures as low as 700 °C to 800 °C [4]. While the hydrogen in gaseous form can readily react with the sample, the solid carbon deposits on the sample first to enable the reduction at higher temperatures. Correspondingly, the TGA shows two subsequent weight losses for the ilmenite sample, 1.5 % at 550-900 °C and 5.5 % at 900-915 °C. A sample that was baked out under nitrogen beforehand shows a similar weight loss of 1.8 % at 700-910 °C and 6.5 % at 910-940 °C. At temperatures above 915 °C and 940 °C respectively, there is strong deposition of carbon, which overcompensates any further weight loss through reduction. The NU-LHT-2M sample did not exhibit a significant weight change under methane besides the expected weight loss due to thermal decomposition of the minerals and the strong weight increase due to carbon deposition above 900 °C. Again the potential side reactions with decomposition products complicate a clear identification of the reduction processes of the sample.

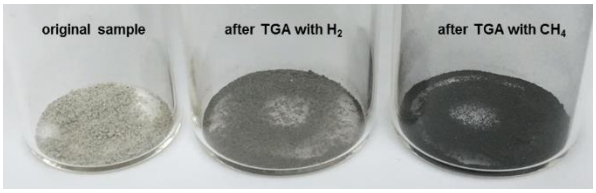

Fig. 1: NU-LHT-2M (100-125 µm) before and after TGA

References: [1] Colozza (1991) NASA Contr. Rep. 189073. [2] Rickman and Lowers (2012) NASA/TM-2012-217458. [3] Reiss et al. (2019), *Planetary and Space Science* (accepted). [4] Chen et al. (1975) *Canadian Journal of Chemistry*, 53(23), 3580-3590.