TALLINNA TEHNIKAÜLIKOOL

INVESTIGATION OF THE HARDNESS AND ABRASIVENESS OF OIL SHALE ASH

Duration: November 2017 – February 2018

Client: ATI PROFIIL OÜ

Analytical Research Facility (ANR):

Tallinn University of Technology,

School of Engineering,

Department of Mechanical and Industrial Engineering,

Research Laboratory of Tribology and Materials Testing

www.ttu.ee/tribo

IReC: University of Tartu

Initial requirements:

Sample type (powders) and sample amount to be provided by client: powder of oil shale ash in required amounts (about 100 kg). Reference material (chalk, talk) in required amounts (about 20-30kg).

RESULTS:

Part 1.

3 fractions of oil shale were separated (0-25um, 25-45um, 45-90um). Close to 20 litres of each fraction of ash were obtained.

Part 2a.

Mohs scratch tests for the 3 fractions of oil shale ash samples were performed. The powder was placed on top of the Mohs set minerals, pressed by hand with force around 10 N and shifted about 50 times from right to left with amplitude of about 5-10 mm in the designated places by cotton pad or by soft rubber. Results are presented in the Table 1-4. Vertical scratches were artificially produced by specially sharpened file to make it possible to observe their removal by moving (in perpendicular direction) of powders. Zones with removed material are shown with arrows.

Table 1. Results with TALC stone (grade 1)

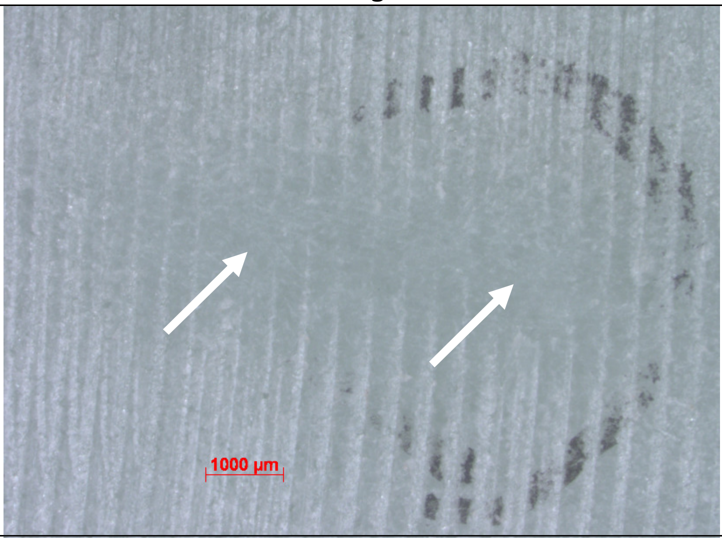
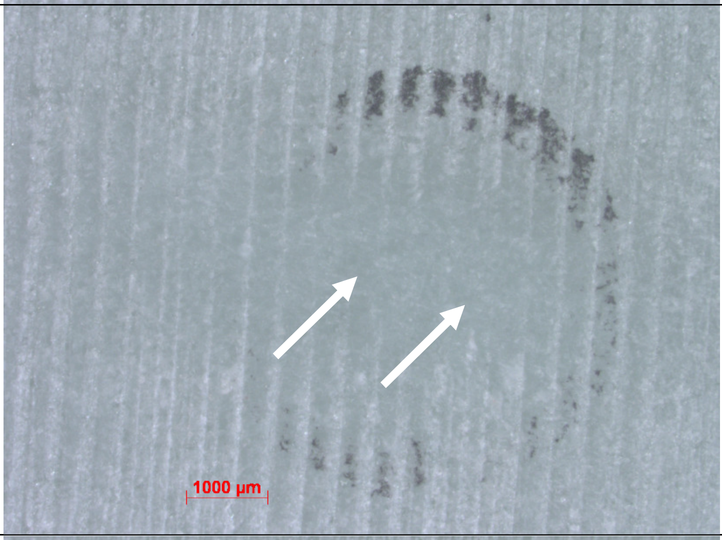
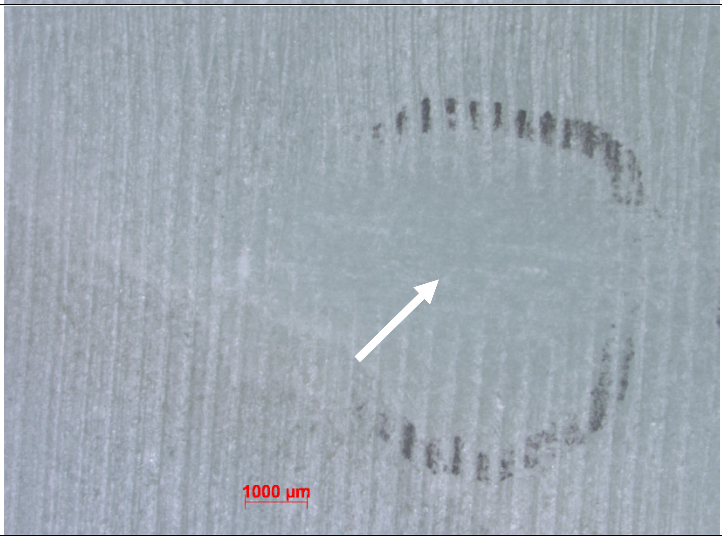
Ash	Image
0-25 µm	
25-45 µm	
45-90 µm	

Table 2. Results with GYPSUM stone (grade 2)

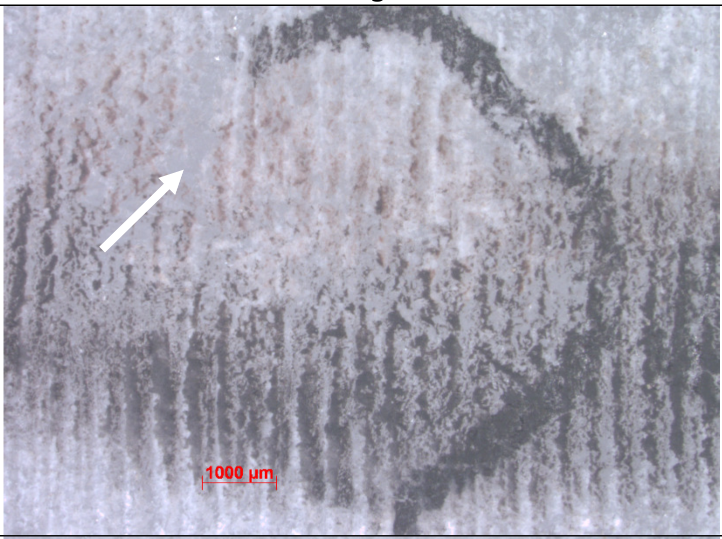
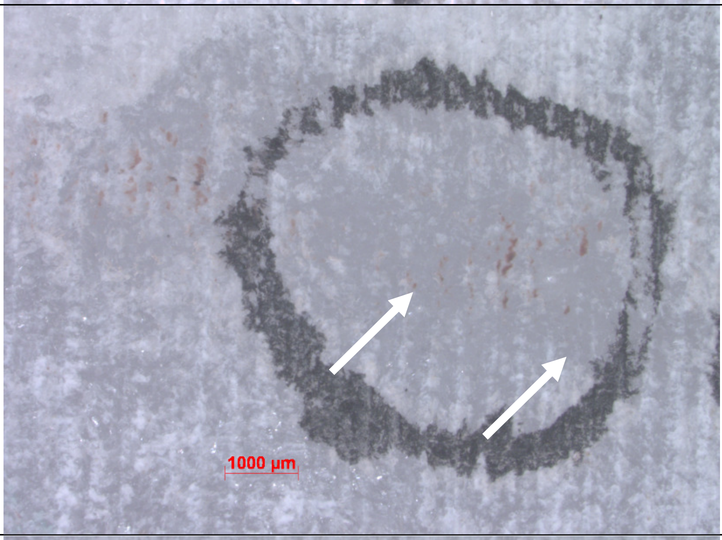
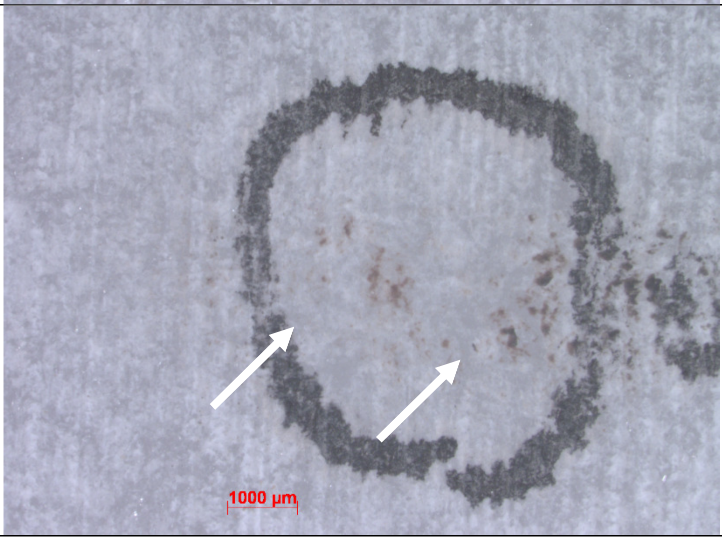
Ash	Image
0-25 µm	
25-45 µm	
45-90 µm	

Table 3. Results with CALCITE stone (grade 3)

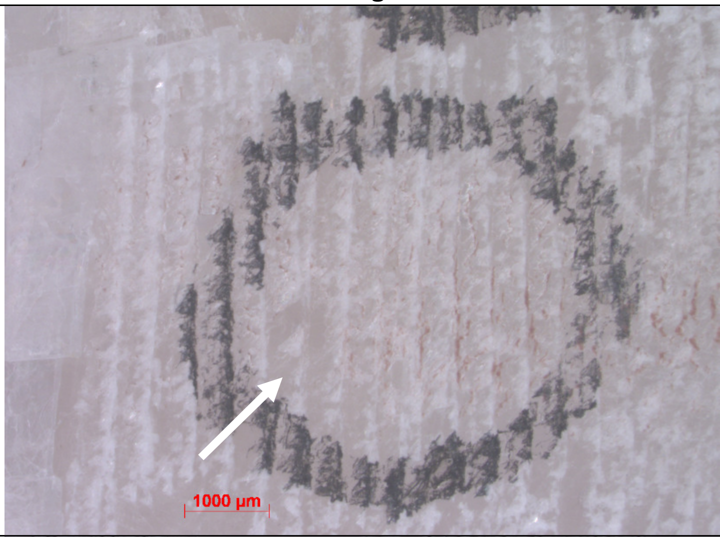
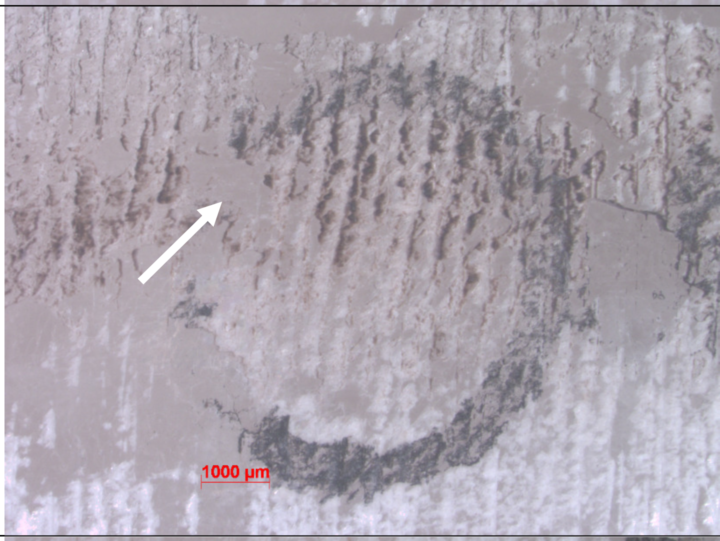
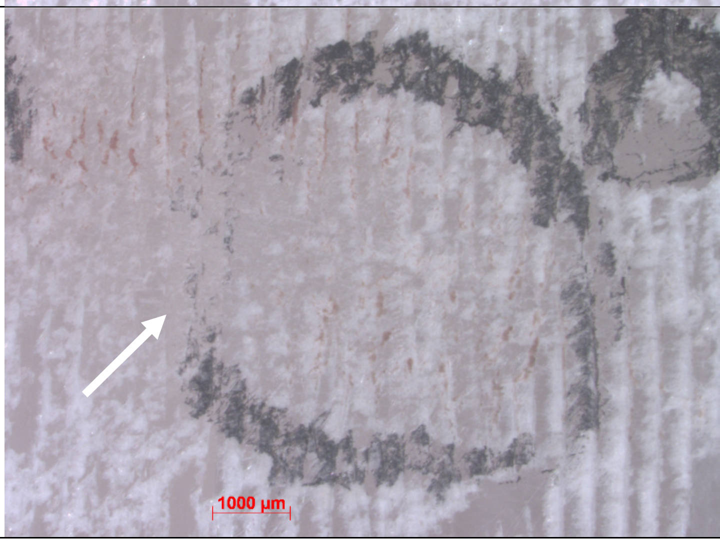
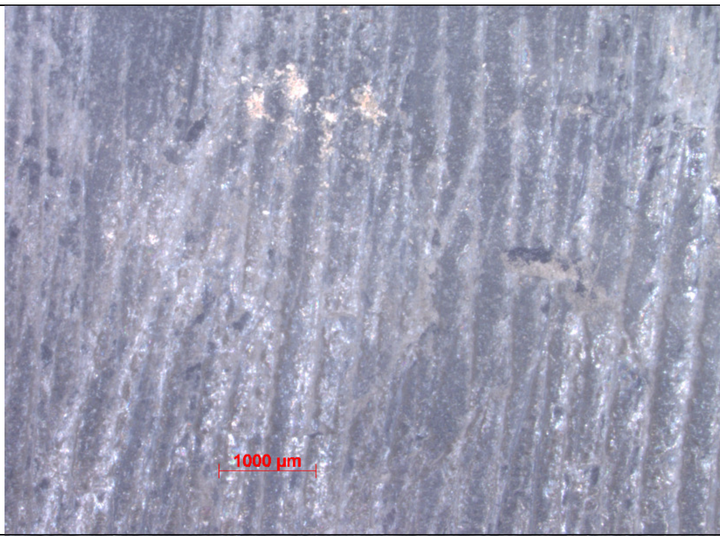
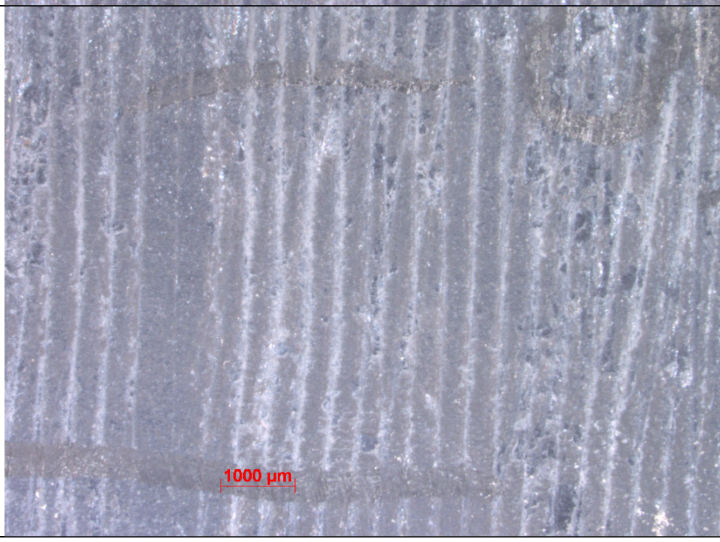
Ash	Image
0-25 µm	
25-45 µm	
45-90 µm	

Table 4. Results with FLUORITE stone (grade 4)

Ash	Image
0-25 µm	Not tested
25-45 µm	
45-90 µm	

Conclusion:

The hardness of ash is more than grade 1 or 2 (according to Mohs).

It is similar to grade 3 but lower than grade 4.

Part 2b.

Sintered ash material hardness testing with Vickers test equipment.

Spark Plasma Sintering device was used to consolidate (make sufficiently strong material) ash powder.

Two tablets with diameter of 20 mm were produced:

Nr 1. Temperature 1200 °C, Pressure 30 MPa, duration of holding at set temperature and pressure was 10 minutes.

Result: The powder was melted and squeezed out of the mould. The pressure and/or temperature should be reduced.

Nr 2. Temperature 900 °C, Pressure 20 MPa, duration of holding at set temperature and pressure was 10 minutes.

Result: Satisfactory tablet (bulk body) was produced. During encapsulation (with the help of melted plastic and average pressure) into plastic (to hold it as one piece during hardness testing) the sample was slightly cracked while some areas were suitable for micro-hardness tests. Polishing with SiC (down to P4000) sandpaper without water was performed to make surface flat.

Result with micro Vickers (load 50 g) showed that hardness is in the range HV0.05 170-280.

According to [1] it is possible to conclude that there is almost perfect fitting between Mohs (MS) and Vickers (VS) scales according to equation:

$VS=(31*MS^2)-(59*MS)+55$

As a conclusion, it could be calculated that HV0.05 170-280 is close to the Mohs 3.1-3.8 (average is 3.45).

Figure 1. Samples produced by SPS from ash.

Part 3.

Part 3.1

Abrasiveness testing for the 3 fractions of oil shale ash (and talc or chalk) with aluminium sample (rectangular 10x10x250mm, Alloy: Sapa 6082-T6 (EN-AW-6082). Surface class = 6. Test zone 10x10x30mm, other part protected by stainless shield cover with thickness of 0.5mm), Figure 2.

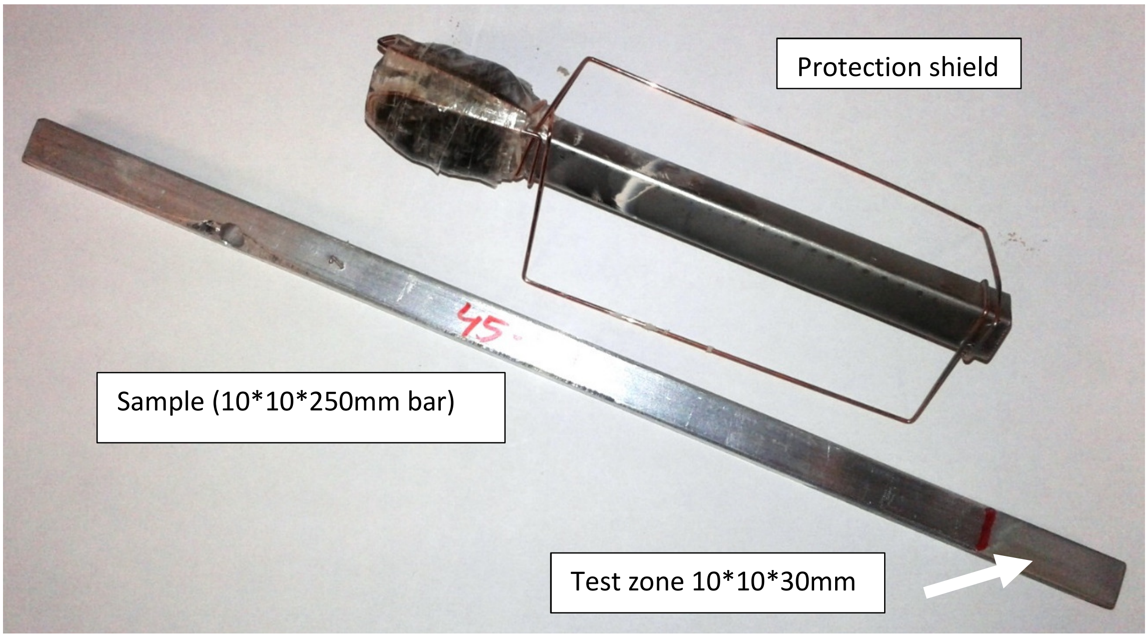


Figure 2. Sample and protection shield separately.

In order to measure abrasiveness, powders were placed into test chamber of device (HLTS, Figure 3) and samples were moved in anti-clockwise direction with radius of 0.12 m with speed of 0,1 m/s. The torque was set as constant to provide constant friction force (resistance to movement) of 31 N for each sample. The maximum stable friction force possible for talc was 7 N and it was used during test. The depth of immersion into the powder was automatically adjusted by controller to provide the required friction force. Proposed duration – 24 hours. Some tests were extended up to 72 hours or even to 312 hour (13 days).

Figure 3. HLTS device

Figure 4. Sample installed into HLTS with indication of movement direction. The sample is not immersed into powder yet.

Main conclusions:

- The mass increase (rise or gain) during testing of all powders was mainly observed. The wear (mass loss) is either very localized or is minor and is compensated by formation of layer composed from powders.
- In some areas of samples, when ash is tested, the wear is visible.
- It was found, that aluminium sample can be scratched during placing and removal of the protection shield (made from stainless steel, especially when the hard powder is accumulated between shield and the sample during test) and this should be avoided to provide correct mass change measurements. It is better to use metallic samples that are harder than shield to avoid such scratching.
- The aluminium oxide formed on aluminium sample can seriously influence the wear resistance of aluminium due to sufficiently higher hardness of oxide (alumina). It is better to use materials that do not form oxides with very high hardness (copper or copper alloys).
- The additional test with preparation of aluminium surface by removal of oxide layer with the help of sand paper or by abrasive grinder were not leading to the mass loss of aluminium sample (i.e. the mass increase was observed).

Figure 5. Typical wear scar after test with abrasive powder (not result of current test).

Figure 6. Results of current testing.

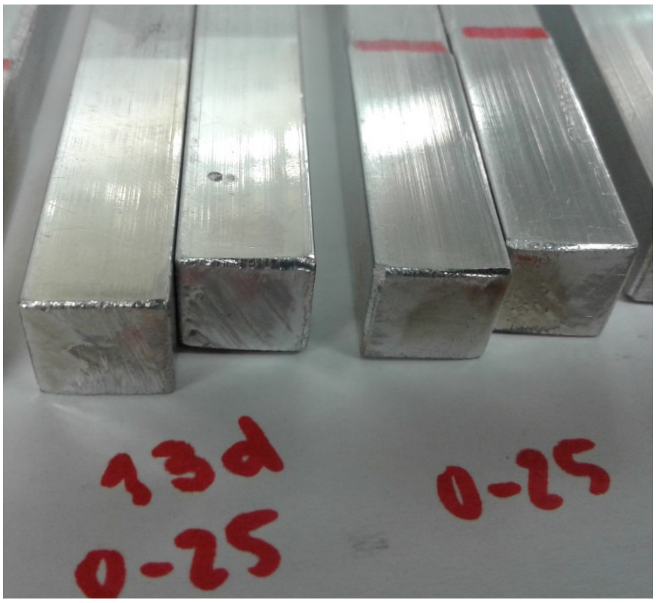


Figure 7. Closer view of sample tested with ash during 13 days (on the left) and two days (on the right). Some polishing is clearly visible.

Part 3.2.

The sample shape was modified to provide higher and more uniform stress. Aluminium “ski” with area of 20x70 mm was pushed against the powder (flat-against-flat contact). The torque was set as constant to provide constant friction force of 37 N. The maximum stable friction force possible for talc was 15 N and t was used during test. Higher friction force was possible to achieve only with chalk.

- Results are similar to those presented in Part 3.2.
- The formation of new surface layers from powder material is more intensive.

Figure 8. Results of testing with “ski” type sample. Clear formation of new surface layer is visible in case of chalk and ash.

Part 3.3

Since it was hard to compare the abrasiveness of the ash, talc and chalk the test according to ASTM G65 standard (procedure B, 10 minutes, force again specimen 130 N, only one test for each powder) was performed with coarse (45-90 µm) ash and silica sand of similar size (Figures 9 and 10).

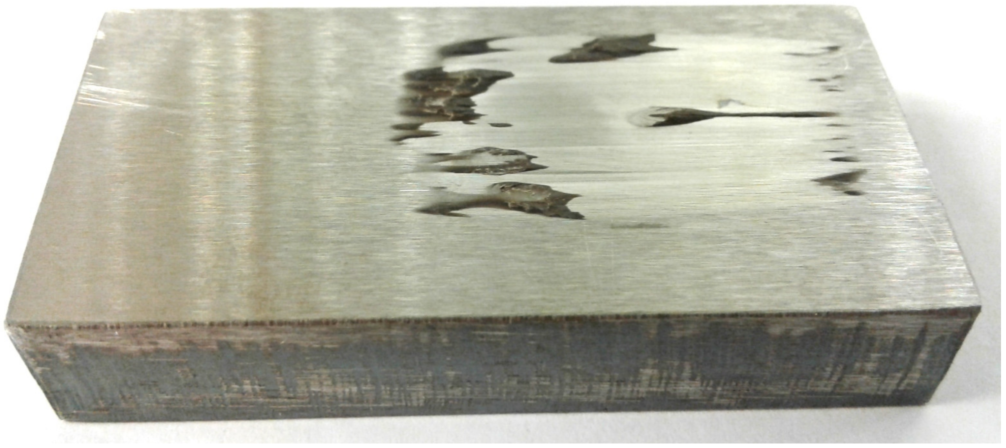


Figure 9. Hardox 400 sample after 10 minutes of abrasion by ash.

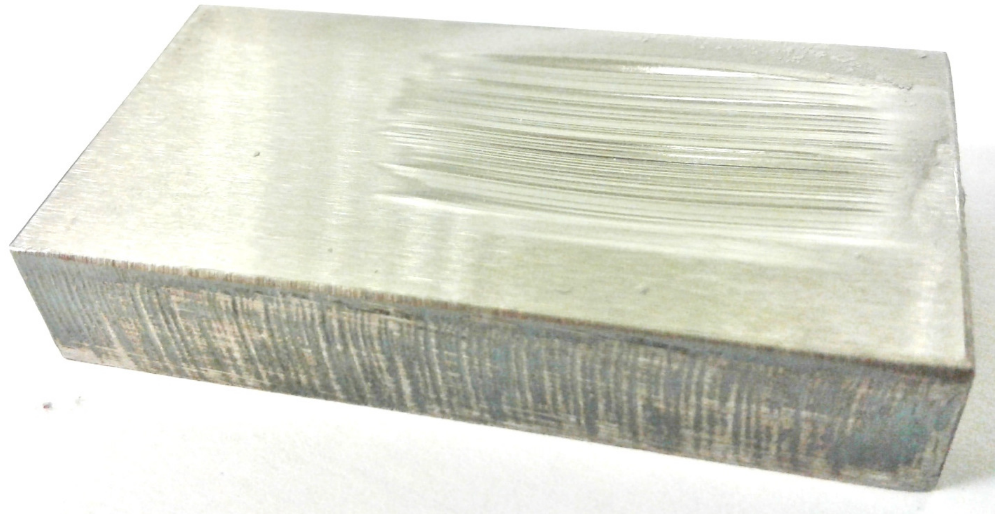


Figure 10. Hardox 400 sample after 10 minutes of abrasion by silica sand.

Mass loss in case of ash was 8.6 mg (without removal of black tribolayer) or 15.3 mg (with removal of black tribolayer) and in case of silica sand the wear was 809 mg. So, the abrasiveness (how well abrasive powder can cut reference material) of ash is at least 809/15.3=52.9 times lower than that of silica sand.

Part 4.

Test result analysis and linking the abrasiveness and Mohs results.

- The conventional Mohs hardness method is not intended for testing of fine powders. However some indicative results were obtained.
- If results of Part 2a and 2b are compared, then it is possible to conclude that hardness of all fractions of ash is close to 3.5 grade according to Mohs scale.
- It was found that finest fraction of ash (0-25 µm) was less effective in removal of artificial grooves but this could be caused by lower effectivity of dragging of such particles by cotton or rubber pad.
- If other material that do not have formation of very hard oxide layer (like alumina in case of aluminium) is used as a sample then probably it will be possible to determine the wear (mass loss) as the action of ash. Alumina has hardness close to grade 9 according to Mohs scale. Aluminium 6082-T6 has hardness HV 95 that is about grade 2.5 according to Mohs scale and mass loss of samples during abrasiveness testing of ash was expected.
- Formation of new surface layer on top of aluminium could be positive (protection of parts) and could be negative (if parts are having very tight tolerances and could be jammed/seized).
- The used test method was applied due to very fine size of powders and due to the fact that they should not be mixed with water. Ash of size 45-90 µm was used to test abrasiveness with the help of ASTM G65 method (rubber wheel abrasion) and it was found to have at least 52.9 times lower abrasivity than silica sand of similar size (coarse ash is cutting Hardox 400 steel at least 52.9 times slower than silica sand).
- Talc is very lubricious powder and has grade 1 Mohs grade hardness. The wear caused by this powder was not expected that was proved during current project. It was not possible to apply same test conditions (same friction force) that confirmed its behaviour as a solid lubricant.

References

[1] https://geology.com/minerals/mohs-hardness-scale.shtml

Complied by:

07/02/2018

Maksim Antonov

Senior Researcher,

Tallinn University of Technology

Other participants: Dmitri Goljandin (sieving of powders, consulting), Fjodor Sergejev (coordination, consulting).