LOW TEMPERATURE TRIBOLOGY AT THE FEDERAL INSTITUTE FOR MATERIALS RESEARCH AND TESTING (BAM)

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Abstract: Friction systems running in cryogenic environment are critical in respect to wear and frictional heat generation because in this temperature range, conventional lubricants can't be applied. In order to test the friction and wear behaviour of appropriate solid lubricants and wear resistant material combinations in cryogenic environment, including liquid hydrogen, special test rigs have been constructed. These devices provide test conditions for tribological investigations at temperatures ranging from 4.2 K to room temperature in liquid or gaseous environment at pressures between 10^{-3} mbar and 20 bar. Investigations on polymers, composites and metals with and without coatings show that there is a broad variety of appropriate materials for cryogenic friction units, but the behaviour of austenitic steels in such systems gives an example of non sufficient knowledge in this field.

Introduction

If tribosystems are operated in cryogenic environment, they are critical in respect to wear and frictional heat generation because conventional lubricants like oils and greases fail at temperatures below about -70°C [1]. By using special lubricants this range may be extended to -185°C [2] but normally in cryogenic engineering solid lubricants or material combinations with low friction and high wear resistance must be employed. An overview on such materials is given by Kragelsky [2]. Polymers like PTFE are widely used in cryogenic engineering and their low temperature properties are reported in several papers [3,4,5]. Kensley and Iwasa [6] investigated the behaviour of polymers as insulation materials for superconducting wires.

Extremely high wear resistance and friction coefficients as low as 0.05 are achieved by surface coating with diamond-like carbon. Some types of such coatings are also suitable for low temperature applications [7].

The most common alloys in cryogenic engineering are austenitic stainless steels because they don't show low temperature embrittlement. In cryogenic tribosystems however, the surface of the materials is simultaneously subjected to low temperatures and plastic deformation. Under these conditions, the fcc FeCrNi austenite can be transformed into the bcc martensite which becomes brittle at low temperatures. This may also be important for components running in liquid hydrogen, because the martensitic structure is sensitive to hydrogen embrittlement too.

Up to now, the development of tribosystems for cryogenic environment was mainly driven by space technology and thus characterised by narrow time scales. Fundamental research and further optimisation has only scarcely been carried out.

In order to investigate the influence of cryogenic environments on the tribological properties of a broad variety of materials, experimental studies in inert coolants are performed at BAM since several years. These studies are currently extended to liquid hydrogen.

Tribometers for Cryogenic Environment

Three different test rigs for tribological investigations at low temperatures are in service (cryotribometers CT 2, CT 3, and CT 4). Two tribometers, CT 2 and CT 3, are designed for solid state friction with samples in a pin-on-disc configuration, consisting of a fixed pin, continuously sliding against the lower face of a rotating disc. Both test rigs are appropriate for measurements in inert and hydrogen environment. In CT 4 oscillating friction in inert media can be investigated. The sample chambers of all cryotribometers are thermally insulated by vacuum superinsulation and cooled by a bath of liquid cryogen or a heat exchanger.

In the cryostat of CT 2 (figure 1) the liquid coolant is filled directly into the sample chamber (bath cryostat). The complete friction sample is surrounded by the liquid cryogen and the environmental temperature is equal to the boiling temperature of the coolant (liquid nitrogen, LN_2: 77 K; liquid hydrogen, LH_2: 20 K; liquid helium, LHe: 4.2 K). The advantage of this method is a very effective cooling of the sample. The frictional heat is not only removed by heat conduction and convection, but also by evaporation of the liquid.

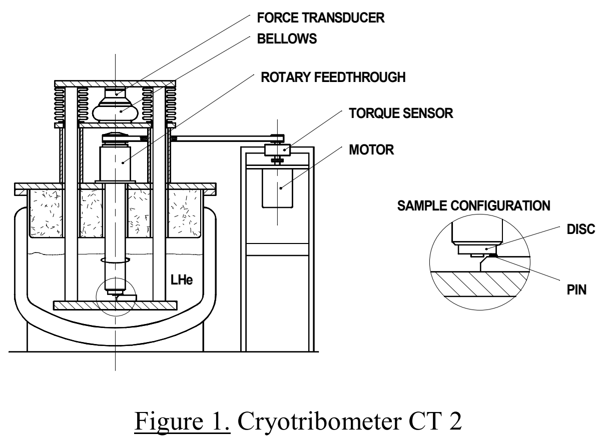

Figure 1. Cryotribometer CT 2

Loading is performed by means of a gas bellows which acts on a frame with the fixed sample (pin) mounted on its lower beam. The mechanical stability of this assembly allows normal forces up to 500 N. The rotating journal is sealed by a ferrofluidic rotary feedthrough at the top flange of the cryostat. The maximum rotation speed is 3000 min^{-1} resulting in a relative velocity of 6 m/s at the friction contact. The friction force can be measured by means of a torque sensor on top of the motor journal or by a beam force transducer similar to the one shown in figure 3.

The tribometer CT 3 (figure 2) can be used as a bath cryostat too, but its sample chamber can also be cooled by a heat exchanger. In this case the sample is surrounded by a contact gas (usually helium) and the coolant is pumped through the heat exchanger, evaporates there and removes heat from the inner vessel (continuous flow cryostat operation). Thus, it is possible to adjust the temperature between 4.2 K (with LHe cooling) and room temperature independently from the pressure. There is no limitation to an equilibrium state of the boiling cooling liquid as in a bath cryostat. The pressure in the sample chamber can be increased up to 20 bar which allows investigations of tribosystems in hydrogen environment above the critical point (critical point of H_2: T = 33 K; p = 13 bar). This is of importance for the design of high performance hydrogen pumps.

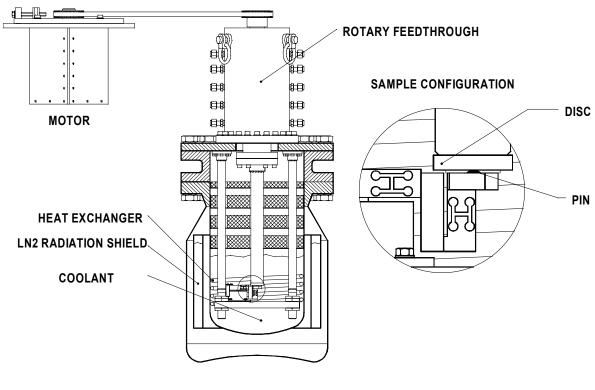

Figure 2: Cryotribometer and bearing test rig CT 3

While in CT 2 the loading unit is at room temperature, in CT 3 loading and force measurement are performed close to the friction couple in the cold part. Therefore, the combined units shown in figure 3, are employed. The sample holder for the counterbody is directly mounted on a two dimensional beam force transducer for measuring the normal and the friction force. Loading is established by pressurized He-gas which acts on a piston that presses a beam with the sample holder upwards against the lower face of the rotating disc. Similar to CT2 the maximum rotation speed is 3000 min^{-1} but the normal force is limited to 100 N for pin-on-disc samples. CT 3 is also appropriate for tests of sliding and roller bearings. In this case the required pre-loading is achieved by pressing two bearings against each other by means of screwed joints.

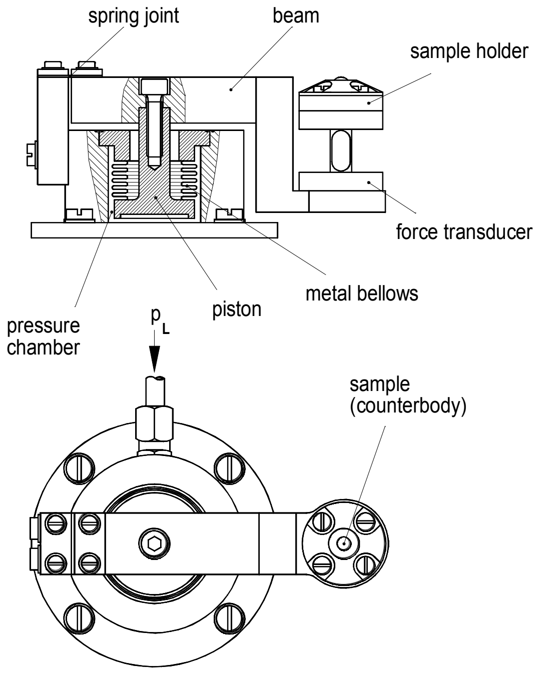

Figure 3: Combined loading and force measuring unit for low temperatures

The tribometer for oscillating sliding CT 4 (figure 4) is integrated in a LN_2-dewar which is also equipped with a heat exchanger for flow cryostat operation. With LHe-cooling and low thermal loads, temperatures close to 4.2 K in gaseous environment are possible. The tribometer insert is designed for loads up to 10 N, a stroke between 0.1 and 5 mm, and a maximum frequency of 10 s^{-1}.

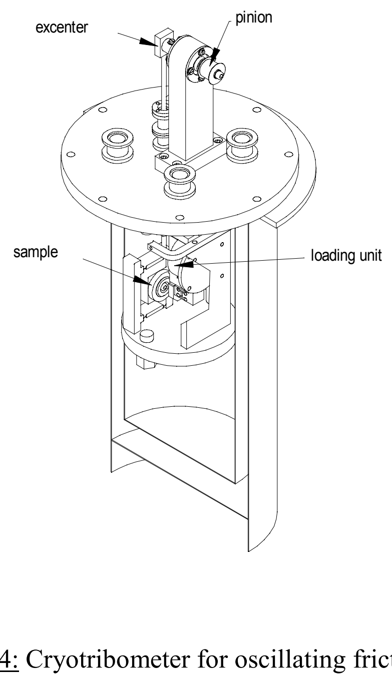

Figure 4: Cryotribometer for oscillating friction CT 4

As standard parameters for sliding friction, a normal force of 5 N, a sliding velocity of 0.2 ms^{-1} and 1800 m sliding distance were chosen. Wherever possible, every material combination is tested under the standard parameters to get an overview over the behaviour of a broad variety of material combination under comparable conditions. For individual experimental programs, the test parameters can be changed within a wide range.

Before inserting the pin-on-disc assembly into the cryostat the sample surfaces are cleaned with ethanol. To remove any residual gases and condensed liquids from the sample chamber it is evacuated to a pressure below 10^{-3} mbar and filled with pure He-gas. The pump-down-refill cycle is repeated three times. During the experiment the sliding force and the displacement of the pin are measured. After the measurement the wear scars of both bodies are examined by profilometry, light, electron and atomic force microscopy.

Results

Up to now, the tribological investigations at low temperatures at BAM are focussed on the behaviour of polymers [8], polymer composites [9,10], amorphous diamond-like carbon (ADLC)-coatings [11], and austenitic stainless steels [12-14] in inert environments like liquid nitrogen and helium.

Polymer materials are of interest because they combine high strength with low density and good tribological properties at low temperatures. The use of these materials is mainly limited by the temperature dependence of their mechanical properties [15-17] which may vary from high strength to viscous flow within a relatively small temperature range. At low temperatures properties like Young's Modulus, yield strength, and hardness are significantly higher which makes materials like PTFE even more wear resistant than at room temperature. A smaller real contact area causes also lower friction. The results of the measurements on several polymers have been published elsewhere [8-10]. Because the polymers with proven good properties in cryogenic tribosystems are not affected by hydrogen environment they are already applied or good candidates for such applications.

To study the stability of the austenitic structure of FeCrNi alloys in cryogenic tribosystems, steel-discs with different compositions (DIN EN1.4301, DIN EN 1.4439, DIN EN 1.4958, DIN EN 1.4591) were tested against Al_2O_3-balls [12-14]. With the exception of steel 1.4591, which did not show any transformation, all tested materials showed an increasing amount of friction-induced martensite with decreasing temperature. At about 20 K a maximum is reached, and at LHe temperature the proportion of transformed material is reduced again. This behaviour agrees with results of low temperature tensile tests [19].

SEM images indicated that the distribution of transformed material corresponds to the distribution of re-transferred wear particles onto the worn surface [20]. Wear debris is the most severely deformed part of the sample surface. It can be stated that martensite generation during tribological stressing depends on deformation behaviour of the material to a greater extent than on the temperature.

After running in liquid hydrogen samples of all materials, including the stable steel 1.4591, showed small cracks in the wear tracks. This indicates that hydrogen embrittlement is not limited to areas which experience martensite transformation. This study will be continued and results of the first measurements are published elsewhere in detail [13].

Amorphous diamond like carbon (ADLC)-coatings have very low friction coefficients and good wear resistance in dry sliding at room temperature. For testing the suitability of carbon coatings for cryogenic applications, several types of single- or multilayer metal containing (Me-C:H-)coatings, some variants of experimental PCVD-coatings (a-C:H) without any additives, and a silicon containing (Si-C:H-)coating were investigated. The coatings had a thickness off 1-10 µm and were deposited on austenitic CrNi-steel discs. The friction coefficient at room temperature was about 0.05 for all coatings. Counterbodies were 10-mm-balls made from AISI 52100 bearing steel or in some cases Al_2O_3.

At cryogenic temperatures considerable differences occurred in the wear behaviour but also during running in. Under all test conditions, the Si-C:H-coatings had the lowest friction coefficient and low continuous wear without catastrophic failure within the test distance, while the other coating types often failed due to rapid delamination. After running in the friction coefficient of the Si-C:H-coatings showed steady state values between 0.05 and 0.3 [11].

In order to test a configuration which is closer to applications, two discs with Si-C:H-coatings were tested in liquid helium in a flat-on-flat geometry. During the tests the normal force was increased from 10 to 25 N and the sliding velocity at the outer diameter from 0.2 to 1.0 m/s. For a sliding distance of 1500 m the friction coefficient was as low as at room temperature (0.05). Then an increasing friction indicated coating failure and the test was stopped. However, the subsequent surface examination showed nearly no wear. Only some small scratches, probably caused by wear debris, were visible. If the wear debris is continuously removed from the contact area it can be expected that these ADLC-coatings keep their good wear protection and lubricating properties for a long duration even at LHe-temperature.

Conclusion

Three devices for friction and wear tests in cryogenic liquid and gaseous environment are in service. Two of them are also appropriate for investigations in liquid and gaseous hydrogen at pressures up to 20 bar. They are used for model tests under continuous or oscillating sliding friction and bearing tests. Investigations on polymers, amorphous diamond-like carbon coatings, and austenitic stainless steels show that these materials are suitable for cryogenic friction units, but the unexpected behaviour of austenitic steels in such systems shows that further investigations in this field are necessary.

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