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Chronology of the microstructure evolution for pearlitic steel under unidirectional tribological loading

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ABSTRACT

The microstructure of the material under the contact strongly influences tribological performance and the ability to control this microstructure therefore is key for optimizing a material's surface properties for low friction and little wear. There is however a significant lack of knowledge about the elementary mechanisms of microstructure evolution under tribological load as well as their kinetics. To cover different stages of this evolution, pearlitic steel pellets sliding against steel 100Cr6 disks were characterized after different numbers of tribological acceleration-deceleration cycles. Scanning electron and focused ion beam microscopy were applied to monitor the microstructure changes. Two tribologically modified surface layers are found: one with bent grain boundaries and one of nanocrystalline nature. We hypothesize that the second layer is formed by breaking down the most bent regions of the first one when a critical grain boundary bending angle is reached and then consumes it from the top.

1. Introduction

In almost all technical systems with moving parts, friction plays an important role [1,2]. In most cases it leads to unwanted energy losses, for example in the piston-cylinder system of an automotive engine [3,4]. The tribological loading changes the microstructure under the contact [5–9]. These microstructural changes in tribologically stressed surfaces influence the behavior of the sliding contact [10,11]. Significant research has been published since the importance of the microstructure in tribology was pointed out [2,7,12]. As early as 1978, Rigney et al. reported highly deformed sub-surface layers in a bronze bearing after unidirectional sliding [13]. Structures smaller than one micrometer were found at the surface with a strained layer underneath [12,13]. To describe and predict these phenomena, a model was proposed for the cause of friction in metals. It focused on the formation of similar sub-surface structures in the steady state sliding regime for metals with like friction coefficients [14]. In later studies, microstructure changes under tribological loading were observed and investigated generally in two patterns. The first concerned itself with changes in the geometry of grains, e.g. grain shape (elongating or bending) and grain size (growth or refinement) [9,15,16]. The second one referred to the changes in crystallographic orientation, e.g. the rotation of grains and sub-grains [17]. As tribological contacting is an extremely complex process, these two patterns could hardly be separated from each other. In tribological experiments on single crystalline copper, elongated cell structures have been found with transmission electron microscopy (TEM) [18]. In a similar system, Kikuchi patterns revealed surface cells rotated around an axis perpendicular to the sliding direction and parallel to the surface [19,20]. Underneath, bent and elongated cells were found [19]. Similar structures have been reproduced with molecular dynamic (MD) simulations [21]. The results above demonstrate that strain induced grain boundary migration can change the shape of grains and simultaneously their crystallographic orientation. There are also works reporting grain refinement and crystallographic orientation changes [22,23]. In reciprocating sliding experiments on copper, the grain size inside the wear track decreased with increasing cycle number [6,24]. A refined surface layer on top of a bent and strained layer was reported frequently [25–30], generally showing a higher micro-hardness than the bulk material [31]. MD simulations indicated the formation of a refined and strained layer [32,33], stating an influence of the crystallographic orientation [32] and interface structure [34,35]. Compared to the MD simulations performed on the formation and development of these sub-surface layers, there is a significant lack of experimental studies in order to understand the basic materials science elementary mechanisms behind their formation as well as the chronology of these mechanisms. The present experimental work focuses on the evolution of the microstructure in an oil lubricated steel-on-steel contact, in which the elongation, bending

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and refinement of the grains in the sub-surface area are investigated in detail.

2. Material and methods

2.1. Sample material

The experiments were carried out using the normalized steel C85 (AISI 1085) for the pin and the hardened and tempered steel 100Cr6 (AISI 5210) for the disks. The diameter of the pins was 8 mm and of the disks 70 mm. The pins were ground with SiC paper of 200 mesh and polished afterwards with 3 μm and 1 μm diamond suspensions (Cloeren Technology, Wegberg, Germany). Due to the polishing process, the pins had a certain, controlled convexity resulting in a maximum height difference of 1 μm from the center to the edges. The ground disks had a hardness of approximately 800 HV, and their roughness was measured to $R_a = 0.1$ μm.

2.2. Tribological testing

In the tribometer (Plint TE-92 HS by Phoenix Tribology, Kingsclere, UK), the C85 pin was pressed with a normal force of 150 N against the 100Cr6 disk, resulting in a contact pressure of 3 MPa; see Fig. 1a for a schematic of the setup. The whole set-up in the unidirectional pin-on-disk tribometer was lubricated with the oil polyalphaolephin (PAO-18) provided by Klüber Lubrication (Munich, Germany) – kinematic viscosity: 4.10 mm^2/s; density: 0.77 g/cm^3, both measured at 100 °C [36]; 125 μl/s oil flow rate – and heated to a system and oil temperature of 100 °C. The distance from the center of the disk to the center of the pin (frictional radius) was 30 mm; the maximum height difference of the disks surface along the frictional radius was 1 μm.

A test cycle consisted of an acceleration to a maximum sliding speed of 2 m/s, a dwell time of 5 s at maximum speed and a slowing down to a complete stop (see Fig. 1b). Each test cycle is referred to as one ramp; half a ramp means unloading the spinning tribometer after half of the dwell time. These ramps were repeated from half a ramp up to 1000 times.

2.3. SEM-imaging

The SEM images were acquired using a focused ion beam/scanning electron dual beam microscope (FIB/SEM), (Helios NanoLab™ DualBeam™ 650 from FEI, Hillsboro, Oregon USA). After applying the tribological stress, the pins were removed from their holders and cleaned in an ultrasonic bath with acetone and isopropanol, ten minutes each. Then they were applied to the FIB-holder with conductive silver and stored under vacuum (< 10 mbar) until the FIB analysis. FIB cross-sections were performed along the sliding direction to get access to the sub-surface microstructure. Before the actual FIB cutting, a layer of electron beam (voltage 2 kV, current 13 nA) deposited platinum was applied to protect the surface from damage due to the deposition of the second layer, the ion beam deposited platinum. This second layer was deposited using 30 kV and 0.77 nA. These two layers were applied to protect the microstructure from damage by the ion beam during milling. Such artifacts were further minimized by performing the cross-section's final polishing step with an extremely small ion beam current, similar to the state of the art technique used in the FIB-based preparation of TEM lamellae [37]. To gain an image, a rough cut was performed using 30 kV and 45 nA, followed by a cleaning cross-section with 30 kV and 2.5 nA to get a smooth surface.

Images of the microstructure were taken via scanning electron microscopy. The voltage and current used for the electron beam during imaging were 2 kV and 0.8 nA. The SEM images were processed with a custom Matlab code. Only the central parts of the images, showing the microstructure of the cross-sections, were manually selected. The platinum protection layers were cropped automatically by creating a binary mask. Then the boundaries between the pearlitic lamellae were detected via changes in the image contrast by using the Matlab-implemented Canny method [38]. On the resulting binary image of the boundaries, a line wise angle calculation was performed. The average bending angle for each horizontal line of pixels was calculated by counting structure elements of two pixels in a 3 × 3 pixel neighborhood and adding up the corresponding polar vectors.

3. Results and discussion

The formation and evolution of different tribologically introduced and microstructurally altered surface layers was examined

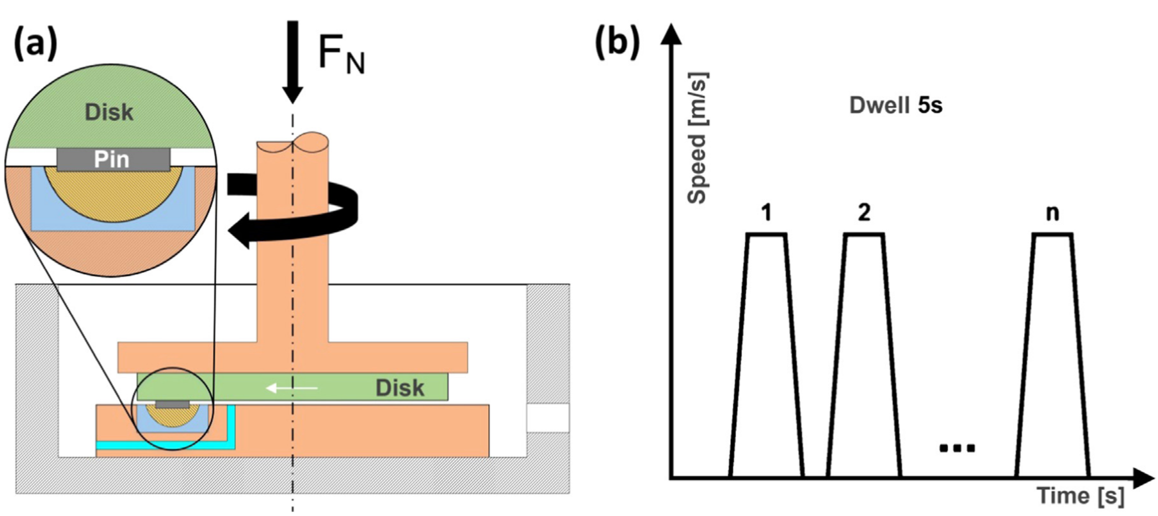

Fig. 1. (a) Experimental set-up of the Plint TE-92 HS tribometer from Phoenix Tribology with the self-aligning bearing for the pin enlarged and (b) the speed profile for the friction tests.