
Structure of surface layers of metal matrix composites

Struktur von Metallmatrixkomposit – Schichten

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Macroheterogeneous composite materials are good prospects for applications as friction pairs. Macroheterogeneous casted composite materials (MCCM) were developed primary for use in severe working conditions such as heavy loads, elevated temperatures, dusty environment, etc., and to replace bronze and other antifriction plain bearing materials. Due to the production technology, secondary nonferrous alloys and even scrap can be used to produce MCCM. These materials are nearly suitable for all friction parts and are unaffected by dust, sand, and water. The structure of MCCM consists of a matrix with uniformly distributed reinforcing particles (e. g., iron based alloys). This feature allows for a reduction in the consumption of nonferrous alloys up to 70%. Depending on the working conditions (sliding velocities, specific pressure, etc.) different compositions of MCCM can be produced. The application of casting technology provides a cost reduction of plain bearings up to 40% depending on the size and design of parts compared to bronze ones. Moreover, in some cases an abnormally low friction coefficient can be achieved and as a result a wearless condition can be obtained.

It is obvious that the service life and performance of bearing materials depend on the processes occurring in the contact area of the friction pairs. So, in order to develop new bearing materials and predict their performance one must have information about the materials' behavior in the contact area.

This paper presents some results on the investigations of macroheterogeneous casted composite materials' behavior during dry sliding of plain bearings.

Makroheterogene Verbundwerkstoffe (composite materials) sind gut geeignet für Anwendungen als Reibpartner. So wurden z. B. gegossene Metallmatrixkomposite entwickelt, die verschiedene Bronzen und andere tribologisch günstige Materialien enthalten, die für solche Anwendungsbereiche geeignet sind und bei denen hohe Druckbelastungen, erhöhte Temperaturen und starke Staub- und Schmutzeinwirkung ertragen werden. Bei der Verarbeitung solcher MCCM läßt sich auch ein erheblicher Anteil an sekundären Metallen und Schrotten mit verwenden. Diese robusten Werkstoffe sind auch für den Einsatz in quarzhaltiger Umgebung bei tiefen Temperaturen und korrosivem Angriff (Wasser) geeignet. Der Gefügebau der Verbundwerkstoffe besteht aus einer Nichteisenmatrix mit eingelagerten Guß- oder Stahlpartikeln, wobei ca. 70% auf den Nichteisenanteil entfällt. Es ist gelungen auf Basis dieser Verbundwerkstoffe Lager zu erzeugen, die ca. 40% billiger sind bei gleichzeitig erhöhter Lebensdauer.

Introduction

By analysing the present approaches used to understand friction processes and wear resistance [1–5], conclusions about the specific and leading role of surface layers in friction pairs can be formed. The change in the physical-chemical activity and the structure of the surface layers (compared to the bulk material) is a reaction to the stress field existing in the friction contact surface.

On the other hand, the degree of the reaction is determined by the chemical composition and structure of phases as well as by the phase distribution and morphology, the interaction between the phases and their interaction with other components in the friction contact region. The wear resistance and friction coefficient are determined by the ability of the structural elements to keep their stability through energy dissipation or through reconstruction (without destruction) into a state which is more preferable for the type of friction. These two mechanisms can occur simultaneously. So, determining the relationship between the initial structure and the surface layers' structure under friction, and establishing the regularities in the appearance of different phases and elements, and

their ability to resist wear, are very important steps in the theory and practice of tribology.

Subsequently many papers have been published dealing with the problem of the surface layers' behaviour under friction, but most papers are devoted mainly to the investigation of steels and cast irons. A multilayer friction surface has been found in ferrous metals. Moreover, this surface is divided into several zones, one of these zones being the "white" layer, consisting of friction martensite as a result of high temperatures and shear stresses [3, 4, 6, 7]. The conditions of crushing and dissolution of primary carbides, the distribution of alloying elements, the degree of $\gamma \leftrightarrow \alpha$ transformations, the character of the dislocation density change, etc., resulting from friction have been established [7–10]. Methods to control wear of structure and the structural and phase changes under different friction conditions were studied as well [11–15]. However less information is available concerning the structural changes in surface layers during the dry sliding nonferrous metals and alloys. These reports mainly deal with friction conditions such as selective transfer [1, 15] or the formation of a "third" body [16, 17]. There are few papers [14, 18, 19] on the structural transformation of MMCs which are reinforced with

rather large ($> 400 \mu\text{m}$) spherical particles and up to date, no study has been published on the structural change in surface layers of such MMCs during friction. The present paper describes the study of the MMC surface layers' transformation during the dry sliding friction.

Experiments

The matrix materials investigated were bronze (CuSi3Mn1) and indium brass LI40 (CuZn40In1.5). Particles ($\varnothing 0.5\text{--}2.0 \text{ mm}$) made of bearing steel (StCr15) were used as reinforcing elements. These materials have been reported to show good properties [19]. As cast materials were machined to obtain friction surfaces with roughness $R_Z = 10$ ($R_A = 2.3$). In this case both matrix and reinforcing particles were in sliding contact. An axis made of 18 Cr1Ti with hardness 55HRC and roughness $R_Z = 3.2$ ($R_A = 0.61$) was used as a contra-body.

The friction tests were performed using the "bush-axis" scheme at specific pressures in the range of 2.0–25.0 MPa and sliding velocities between 1.0–12.0 m/s. Dry friction was used for pressures up to 10 MPa. During and after testing, the total gap between the axis and the bush was measured. The wear was determined after tests according to standard method [1]. The bushes were cut into specimens for microstructural investigations normal and 45° to the surface. For microstructural analyses specimens were etched using FeCl_3 ,

Cr_2O_3 (with Cl ions) and Grosbeck reagent. Microhardness was measured at a load of 20 g. In the thin layers the microhardness was measured according to ref. 28. Optical and electron microscopy were used for the study of the microstructure as well as Auger – microscopy.

Composite's structure

The macrostructure of the composites consists of a matrix with rather uniformly distributed reinforcing elements. The structure of iron particles is fine pearlite. The bronze matrix has the grain size 30–60 (μm) (Fig. 1a). During the formation process the matrix reacts with the iron granules and is enriched with iron (up to 1%) and chromium (up to 0.3%), but loses the manganese (up to 0.5%) and silicon (up to 1.8%). There is no free iron or chromium in the matrix. Manganese diffuses into the iron granule and diffuses from the surface to the centre. Silicon interacts with the iron and forms a thin interlayer of silicides. The copper does not diffuse into the granule but due to the nonequilibrium crystallisation conditions, the deviation from the nominal copper composition is 5–8%. The microhardness of the iron particles is 225–240 HV and the matrix is 95–110 HV.

The brass matrix has a weakly developed dendrite structure (Fig. 1b). The iron granules do not interact with the matrix during the formation process. Dendrites of the matrix consist

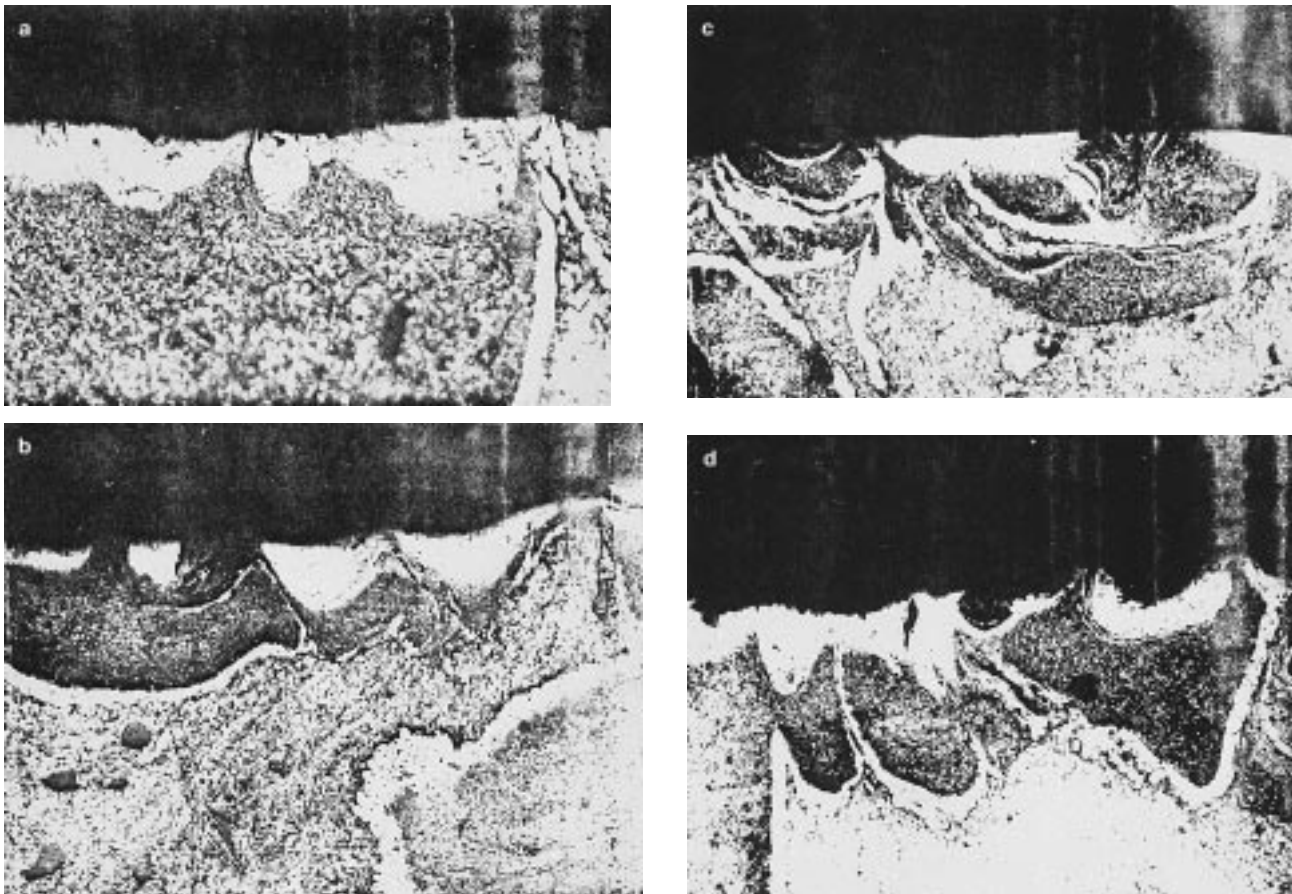


Fig. 1. The formation of films on the surface of the composite material with bronze matrix: a), b) – on the granule surface; c), d) – on the matrix surface ($\times 400$, inclined cross-section)

of 68–72% Cu, 27–29% Zn, 0.2–0.9% In. The interdendrite composition is 56–58% Cu, 38–42% Zn, 2.0–3.2% In. The microhardness of the reinforcing particles is 200–220 HV, the microhardness of dendrites is 90–95 HV, and the microhardness of the interdendrite layers is 120–130 HV.

Testing results

During testing all specimens reveal a similar reaction to the load and the velocity. There is a decrease of the frictional work and the surface temperature for some period of time, followed by a stabilisation of these parameters with a further increase in their values. Thereafter the cycle repeats again. With increase in loads and sliding velocities the duration of the stable work decreases as well as the cycle amplitude. But the leveling down of parameters does not take place for all ranges of loads and velocities, nor does the cycle amplitude reach its initial value. In general, a first approximation of the cycle amplitude can be expressed by the equation:

$$\gamma(x) = A_0 \cdot 3.3^{-\frac{1}{b} \cdot \tau} \cdot \sin(\omega x + \varphi_0)$$

where: A_0 , φ_0 – constants depending on the load and the sliding velocity and ω – frequency determined from the equation:

$$\omega = \omega_0 \sqrt{1 - \left(\frac{3.3}{\omega_0}\right)^2}; \omega_0 - \text{initial frequency}$$

The amplitude decrease during the damping time $\tau = 1/b$ is equal to 3.3. The main influence on the frequency increase and the amplitude decrease is the velocity, and the applied load shows a smaller influence. It was found that composite materials with the brass matrices are characterised by a higher amplitude change of the friction work.

The measurement of the gap between the friction pairs reveal that the gap distance increases during decreases in the frictional work. Then after several cycles the gap either increases or decreases in value. For the specimens the gap increase is greater for larger sliding velocities (ranging from 12 to 25 m/s). There appears to be a direct dependency between the gap and sliding velocity for the range mentioned above. Sixty percent of the friction pairs tested show a stabilisation of the gap value or even its decrease compared to the initial one. It has been established that the connecting diameters periodically increase and decrease.

The roughness of the axis and the bush decreases with testing time. After the tests the roughness of the bushes and axis are $R_z 0,2-0,3$ ($R_a - 0,05$) and $R_z 0,18 - 0,25$ ($R_a 0,003$) respectively. One can compare these values with the initial ones (see above). A typical change in the bush surface during the tests is shown in Fig. 2.

After etching of the specimen an etch resistant area is seen the surface layer. The thickness of this area is 10–900 μm . The growth of this area starts at rough protuberances and then spreads both along the friction surface and inside the composite material. For a certain period of time (depending on the load and the velocity) the film is destroyed and then reforms (Fig. 3). It divides into several layers that exhibit different ability to be etched. With growth of the layers, sections of the film move towards the material's center and become smoother. It can be easily distinguished on the microstructure

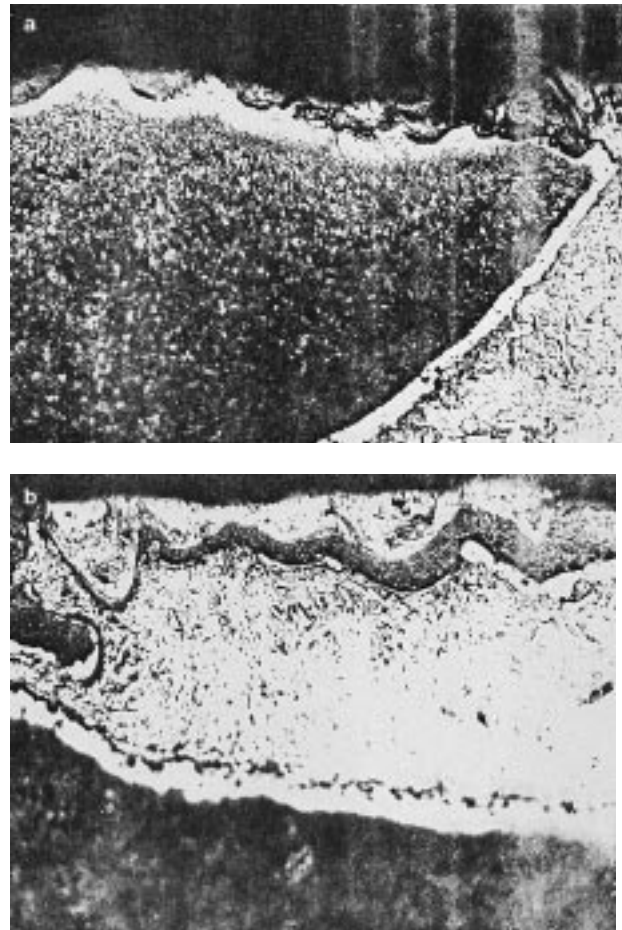


Fig. 2. The friction film on the surface of the composite material with bronze matrix: a) – on the granule surface; b) – on the matrix surface ($\times 400$, inclined cross-section)

of the brass (LI40) based composite material (Fig. 4). Small areas like liquid drops under the film were found in LI40 based composites at loads > 10 MPa and velocities > 5 m/s (Fig. 4d).

The microhardness of the granules and the matrix remain unchanged during the tests, the deviation being less than 20 units. But for all specimens (without exception) a sharp increase in microhardness is observed within the first layer of the friction film (the layer which lies most closely to the matrix and granules). In layers above the granules the microhardness is 360–480 HV and above the matrix the microhardness is 290–310 HV. An additional layer with microhardness 36–42 HV is found in specimens with films thicker than 30 μm . This layer intersects the friction surface.

In the formed hard layer the iron content is 90–95%, 1.9–2.6% chromium, and 0.6–0.9% manganese. The silicon content depends on the matrix material. CCMs based on the bronze matrix are characterised with 0.4–0.6% silicon content in the hard layer, but there are also areas with a silicon content up to 3.2% and manganese content up to 5.2%. As for the CCM with brass (LI40) matrix the average silicon content in the hard layer is 0.3%, but there are areas with a silicon content up to 2.9% and manganese up to 5.4%. In this layer the copper content is less than 0.9%.

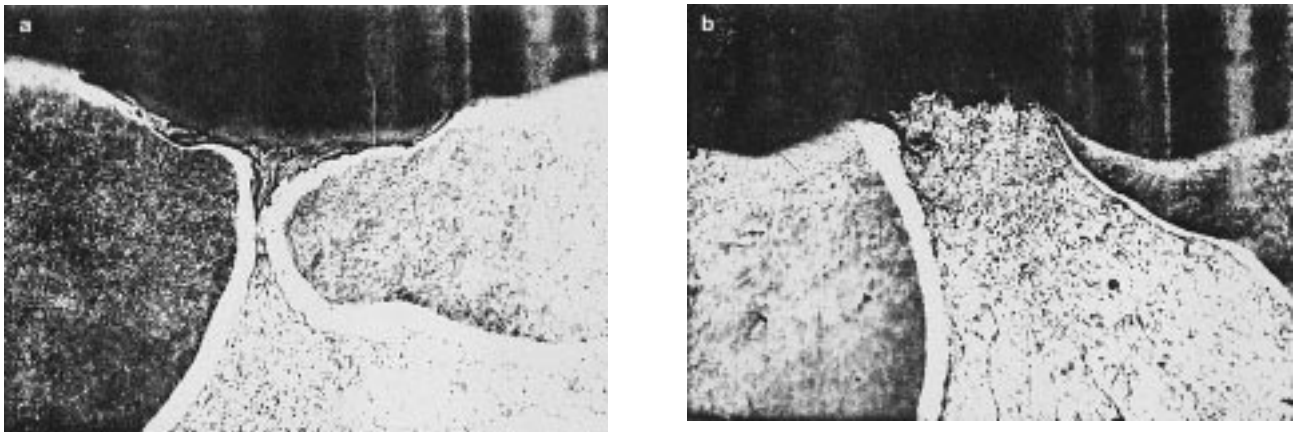


Fig. 3. Destruction of the film on the surface of the composite material with bronze matrix:
 a) – from the granule surface; b) – from the matrix surface
 ($\times 400$, inclined cross-section)

The analyses show that for practically all specimens copper is only present in the very upper surface layer. The concentration of copper decreases with increasing distance away from the surface towards the bulk material. But in deeper layers larger amounts of iron and carbon are observed. There are

also no traces of oxygen in the surface layers, but it is found at depths more than 1000 \AA . Depending on the initial film the oxygen concentration first sharply increases and then abruptly decreases. The thickness of the layer in which the maximum oxygen concentration drops to a minimum value does not ex-

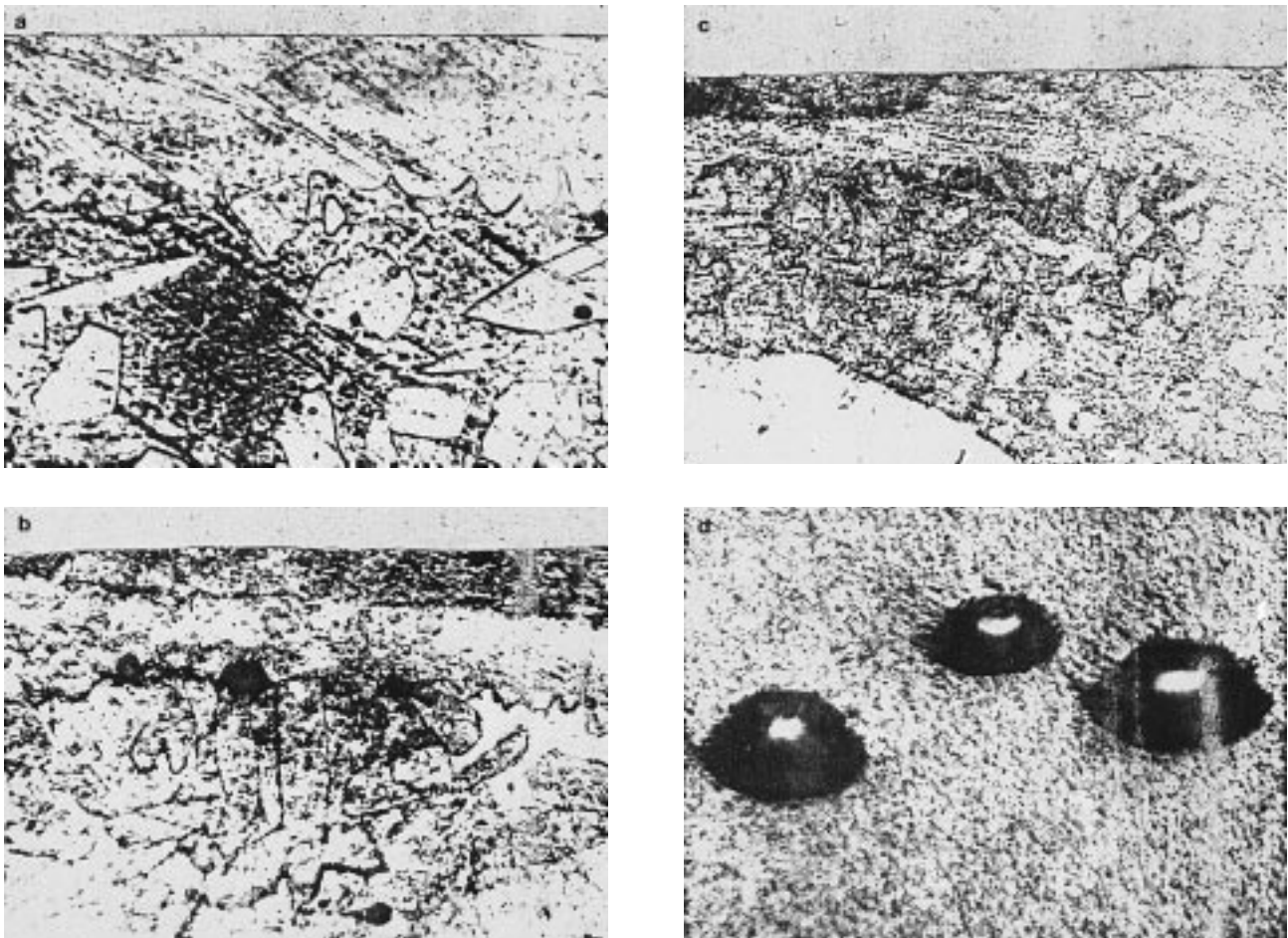


Fig. 4. Friction film on the surface of the composite material with brass matrix:
 a) – on the matrix surface, b) – on the granule surface; c) – film on the transition surface from the matrix to granule,
 d) – liquid drops in the film;
 (a, b, c – $\times 850$, d – $\times 3000$, inclined cross-section)

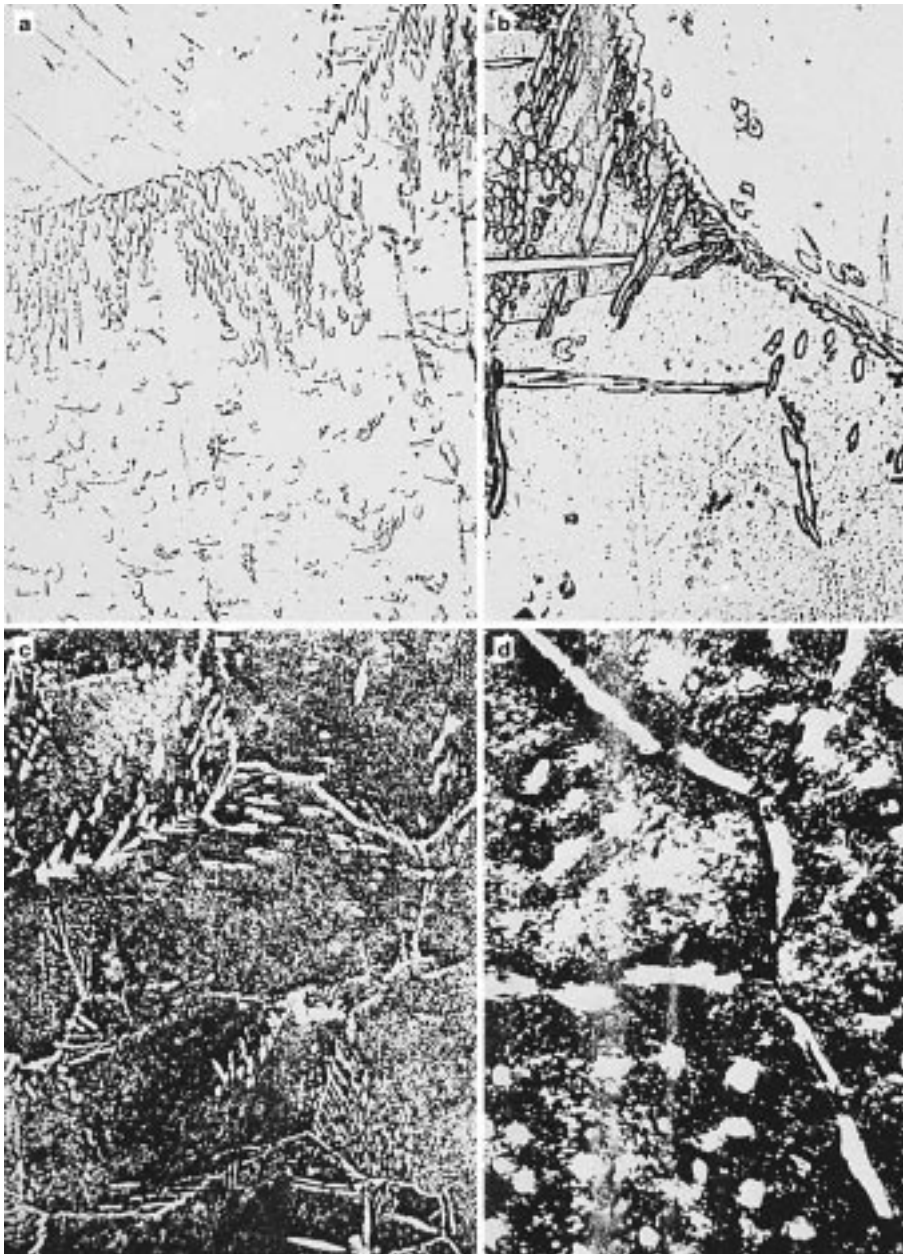


Fig. 5. Destruction of the austenite grains in the friction film:
 a), c) – composite materials with bronze matrix; b), d) – composite materials with brass matrix: ($\times 3000$)

ceed 3000 \AA . Analysis of a phase spectrum leads one to conclude that copper oxides of a lower valency are present in the upper surface layers. In deeper layers copper oxides of a higher valency and Fe_3O_4 are found. Moving from the surface layer to the bulk, one observes a decrease in the copper oxide concentration and an increase in of the iron oxide amount. Chromium, silicon and manganese are not found in the surface layer. The structure of the hardest layer is the same for all composite materials studied. It probably forms as a result of austenite decay with a primary liberation of carbides (Fig. 5). In layers situated close to the granule surface areas enriched with carbides are observed (Fig. 6). In the horizontal direction, towards the matrix areas, a mixed structure containing austenite and martensite has appeared. A long the vertical direction, toward the surface, the structure within the most hard layer changes from bainite through the trostite to sorbite. Sorbite pearlite is the base of the first area of the less hard layer and towards the surface structural elements be-

comes more coarse. Simultaneously copper particles are found in the structure. Copper or a solid solution based on copper penetrates along boundaries into grains of the iron based phase. A new structure forms (Fig. 7) where the copper concentration increases towards the friction film's surface. In the layer enriched with copper a very deformed structure is observed at the first stage of applied friction. This structure is replaced with one typical for secondary recrystallisation, i.e., the growth of large grains. On the surface of these grains lines similar to twins are indicated (Fig. 8). This structure is changing as well. New grains appear, raised slightly above the initial grains. These grains form a new phase which differs considerably from the ones observed before. It is worth noting that the grain coarsening of this new phase is not similar to that of primary not secondary recrystallisation. These phases are porous and many of the pores have an edged shape similar to reverse crystals. It may be evidence of the pores' formation as a result of the migrating vacancies. To identify the structure

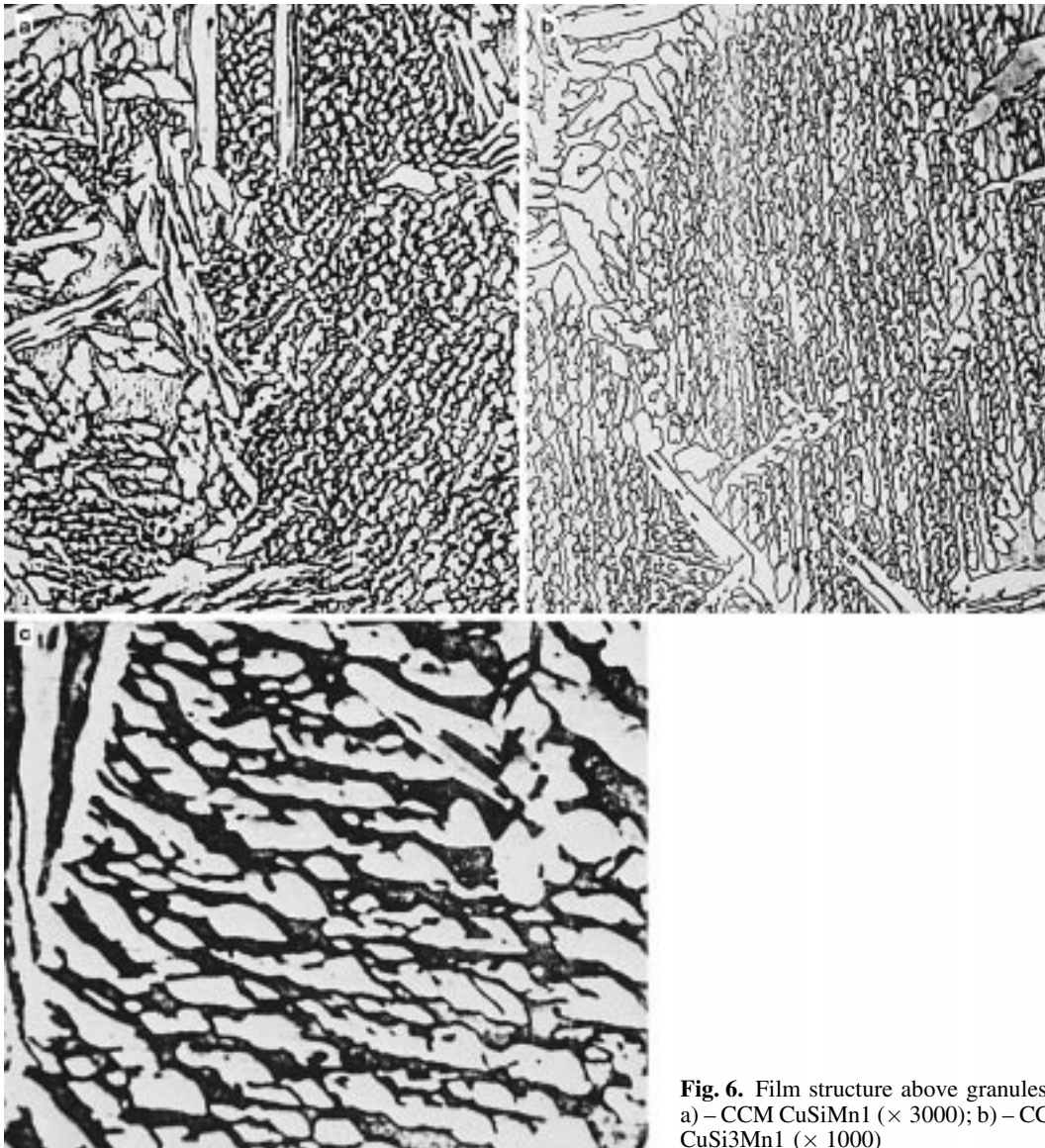


Fig. 6. Film structure above granules:
a) – CCM CuSiMn1 ($\times 3000$); b) – CCM Li40 ($\times 3000$); c) – CCM CuSi3Mn1 ($\times 1000$)

TEM microscopy was applied (Fig. 9). Grain boundaries can be clearly seen. Moreover, there are some pores in the grains. Some grains merge and become coarser without a clear boundary, perhaps as a result of finishing with ions. The initial structure contains defects and a considerable number of dislocations. One can note the presence of edged pores both in the matrix and in the grains of a new phase. The average grain size of the new phase is about $1.6 \mu\text{m}$.

Discussion

The formation of the friction film on the surface of composite materials is likely the result of a normal wear process (at the initial stage) together with an abrasive wear and transfer of the second kind. Normal wear is accompanied by intensive plastic deformations within a thin surface layer with a distribution of accumulated ultrafine wear products. Taking into account the small duration of the first stage we can assume

that abrasive wear makes the main contribution, the reason being the considerable difference between the hardness of the contra-body and bush (up to 2.5 times). High loads and velocities assist the transfer between the contrabody and the bush. The fast moving of wear products into the zone of the film formation is indicated by the velocity of film formation, phase and structural compositions. The film is a structural body with several layers and transition areas between them. Each of these layers has a different structure and phase composition. The initial material for the film formation can be only products of triboconnection wear. The consolidation of separate particles (even taking into account their microsized and structural defects) can take place only at high temperatures. Taking into consideration the time factor of film formation the minimum temperature threshold can be estimated as a temperature equal to 0,3 the melting temperature of the low melting component. A comparison of data obtained shows that the low melting component can be a copper based solid solution, which is a common phase in the composition of both composite materials investigated. The austenite formation

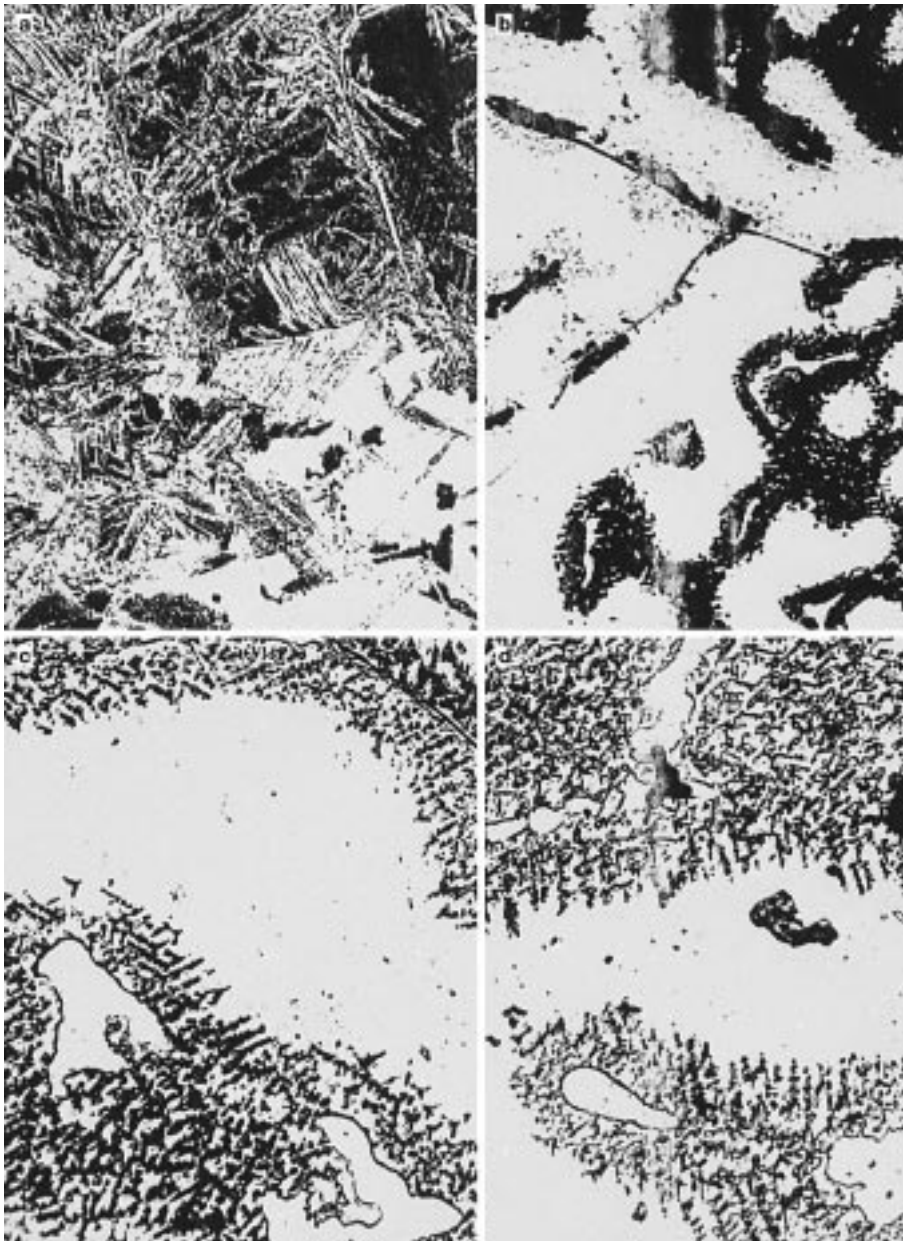


Fig. 7. Film structure above granules: a) $\times 1500$, b) $\times 3000$, c) $\times 10\,000$.

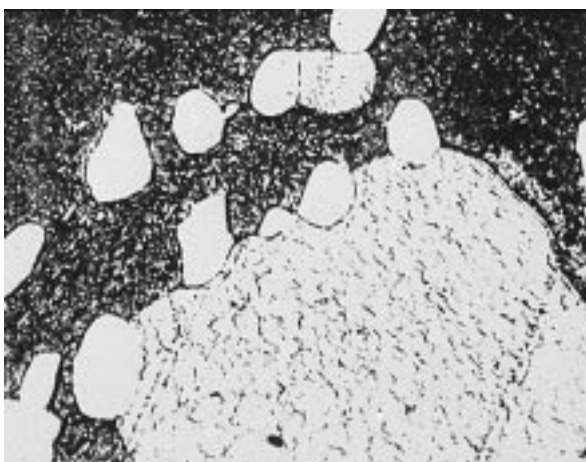


Fig. 8. Recrystallisation in the friction film of CCM Li40 (upper layer, $\times 10\,000$)

is impossible at temperatures below $723\text{ }^{\circ}\text{C}$. The dissolution of carbides takes place at even higher temperatures. The friction film forms during the sintering and contact-reactive melting processes. These processes are characterised by a strong diffusion of elements. It can be assumed that in the initial stage of film formation contact-reactive melting plays the dominant role. This situation arises when there is either a system with a minimum in its solidus-liquidus curves or an eutectic system. The second requirement in this melting process is a certain minimum temperature, and the third requirement is an active contact surface. It is known [20] that the contact melting temperature decreases under nonisothermal conditions and high pressures. The lowering of the contact melting temperature is larger when the heating rate considerably exceeds the rate of nucleation of the chemical compound in the eutectic system. The contact melting process is intensified when thin oxide films are present in the contact zone. Especially the melting process proceeds rapidly at the “oxide film-me-

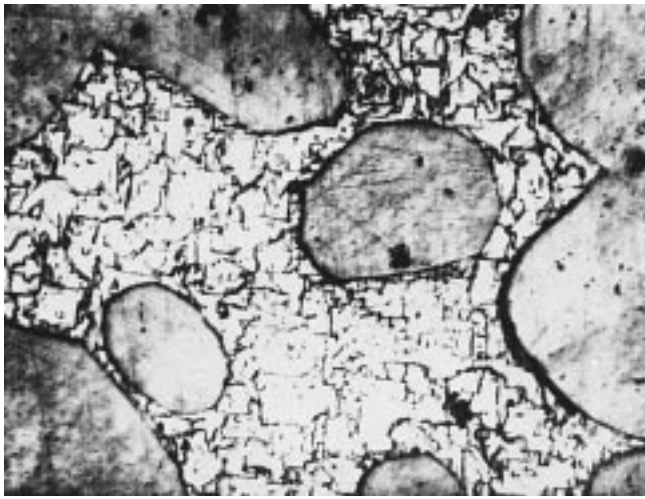


Fig. 9. Copper precipitation on the surface in the friction film of CCM CuSi₃Mn₁ ($\times 900$)

tal” interface. In this case the disintegration and melting of the oxide film take place without the overheating of the liquid phase. And lastly, to realise the contact melting process it is necessary to fulfill the energetic requirement, i. e., the system must have sufficient amount of activated atoms at the interface of contacting phases.

The systems studied meet all of these requirements completely with respect to composition, temperature increase arising from mechanical energy dissipation, and energetic state (ultrafine particles are very active).

Further evidence of the initial particles’ contact-reactive melting is the presence of structure areas with an abnormal concentration of elements. The composition of the interlayer in the liquid and solid state is an invariable. There is neither a redistribution of elements through the liquid interlayer nor the formation of solid solutions. It can be explained by the higher dissolution rate compared to the saturation rate in the solid state. Due to overheating the liquid interlayer can be enriched with a low temperature component which forms an eutectic or minimum on the liquidus-solidus curves. As a result the concentration of the composition can vary within 10%. In the zone of the interactions between steel and copper particles the concentration practically reaches the maximum value for the iron based solid solution. The carbon concentration slightly increases in this zone. All this together proves the presence of the molten copper. The last is proved by the specific interaction (copper penetration along the grain boundaries and inside grains) as well [21]. The probability of such a diffusion interaction in the solid state is negligible due to the very short time period of film formation.

After the film’s formation the friction job sharply decreases and the friction conditions of work become easier. In lower layers (containing mainly steel) all changes run according to the scheme of fatigue fracture. Another picture can be seen in layers containing copper. It is worthwhile to pay attention to secondary recrystallisation. It is known that this process can proceed within a narrow range of rather high temperatures in order to stabilise an initial recrystallised matrix; moreover, there must be grains available for growth. In the materials the dispersed oxides such as Cu₂O, CuO, FeO, Fe₃O₄ cannot stabilise the matrix, as these oxides are distributed locally and only in small amounts. Moreover,

they are not available for dissolution. It is necessary to note that there are no others impurities in sufficient amounts to stabilise the matrix. Probably, the matrix’s stabilisation is a result of the structural inhibition which plays the leading role in the recrystallisation of copper.

The next change in the structure and phase composition of the film’s surface layer takes place during a stable friction regime. This change deals with the destruction of the secondary recrystallisation’s structure and the formation of a phase with a high copper concentration. The form of the obtained phase is similar in character to the copper or its alloys’ surface which have been subjected to corrosive influences. As a result the reverse deposition of pure copper then takes place [22–24]. For all cases of corrosion interaction the deposition of a pure copper phase involves electro-chemical reactions at a cathode (copper) and anode (electrically more negative element). The copper, defined as the cathode, results in the ion’s appearance as mono- or di-valent copper ions which serves as a carrier of electrons from the metal to the oxygen. The products of copper oxidation are reduced on the anode causing the oxidation of its material. The oxidation rate (copper corrosion) is controlled by the rate of the cathode oxygen reduction. First, the dissolution of more electronegative elements takes place. For the alloys it means the appearance of a layer with a high concentration of nonequilibrium vacancies and enriched with copper. The high vacancy concentration defines the large values of the diffusion coefficient and copper atoms’ activity. All of this provides the conditions necessary for the copper ions to be reduced to metallic copper within its own phase. Copper ions are reduced on the surface of a dissolving material resulting in the formation of the pure copper surface layer. This layer consists of very fine crystals (0.09–1.2 μm). Moreover, this layer is porous and does not decelerate oxidation-reduction reactions. With the increase of the concentration of an element-anode the vacancy concentration (due to the anode oxidation) grows faster resulting in more favourable conditions for copper to form within its own phase. For the multiphase alloy, when one phase is more electronegative, the process of copper precipitation is matched with the additional work of the element “positive phase-negative phase”. This work assists the concentrated growth of the copper oxidation products and as a result, the rate of the electrons transfer by the copper ions from oxygen to the corrosion surface is increased.

The similarity in the structure and phase compositions between the surface formed by the reverse deposition of copper ions during corrosion and the upper layer of the friction film allows one to suppose that they share similar formation mechanism. This similarity is proved by the same shape and sizes of grains and their coarsening with time, where considerable number of vacancies merge into pores, the absence of linear defect traces, and low hardness. Thus the formation of the upper friction layer on the surface of composite materials studied can be assumed to result from electrochemical oxidation-reduction reactions during the diffusional displacement of material components. This assumption is supported as well by the electrochemical process of copper oxidation in atmosphere conditions [22]. The conditions for making this mechanism possible are the electrical positive element (copper) and electrical positive elements (Zn, Cr, Fe, In), and the strong reducing agent (carbon). Structural elements of the friction film endure influences resulting in the distortion of crystal lattice and formation of structural defects. It causes the formation of complexes where vacancies migrate very easily. A stressed state of the material influences the character of interaction

processes between the friction surface and the media accelerating the sorption and electrochemical interactions [26].

The temperature factor assists the electrochemical reactions as well. The formation of the film upper layer begins at the lowering of the friction work resulting in a decrease of the temperature at the contacting surfaces. But there is some surface heating which does not reach the equilibrium with the surroundings. Electrochemical properties of copper crystals' edges are notably different. This difference is defined by the lattice distortion in one of the crystallographic directions. So, all the important conditions for the electrochemical reactions for the reverse deposition of copper ions during the formation of the friction film's upper layer are realised.

It is worth noting that some properties of the copper layer of the friction film are similar to ones of servovite film. First, there are similar structural features: microporosity, residual edged pores-reverse crystals, the absence of linear defects, oxides on the surface, etc. Secondly, these films are not suitable for work hardening and are characterised by a low resistance threshold to loads and very small shear forces [1, 15]. The formation of servovite film follows different mechanisms depending on the lubricants, friction materials used, and working conditions. But the necessary condition for high film activity is the reduction of copper oxides [1, 15, 27].

Conclusion

The friction film is produced on the composite materials' surface independent of the formation conditions and a low friction coefficient. This film increases the service life of the friction pair during dry friction within a wide range of velocities and loads. Moreover composite materials developed can be used in conditions unsuitable for common anti-friction materials. The question whether the film formed is a servovite film or not remains unanswered.

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