ABRASIVE/CORROSIVE WEAR ON PLASTIC MOLD STEELS, MEASURED UNDER PRACTICAL PROCESSING CONDITIONS

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For testing wear on plastic mould steels, experimental results of two testing devices will be discussed. The apparatus with integrated measuring technology allows wear measurements under practical injection molding conditions. This apparatus is operated as injection mold in a commercially available injection moulding machine. All of the wear relevant values can be measured with integrated temperature- and pressure-sensors. Wear is detected by weight- or volume-loss measurement of the specimens and is plotted as the mean depth of abrasion (Δh in µm) dependent on the abrasion work per surface unit in the gap in kJ/cm^2 according to an energetic wear model. The second testing device presented is a new practical laboratory test under boosted corrosion conditions. The apparatus is operated like a plate-plate rheometer at temperatures between 180 °C and 300 °C, rotational speeds up to 650 rpm, shear rates up to 5,000 s^{-1} and measuring times between several hours and some days. High temperatures and a combined dynamic/static testing mode are used to accelerate the degradation of the polymer and its additives.

Introduction

In plastics processing machinery and moulds, wear occurs primarily as abrasion on screws, cylinders, non-return valves and in the flow channels and cavities of the moulds [1-3]. Wear diminishes the quality of the injection molded part and the semi-finished extrusion part, for example by variations in size and weight and discoloration and leads as well to higher replacement parts and maintenance costs and to clear reductions in service life [1]. For melt-conveying machine parts and moulds, steels are needed with excellent wear and corrosion resistance [1-3, 5, 6]. The aim of a joint project of the Institute of Plastics Processing at the University of Leoben and Boehler Edelstahl GmbH, Kapfenberg, Austria, was to develop a new wear testing apparatus with integrated measuring technology in order to measure all wear relevant process parameters.

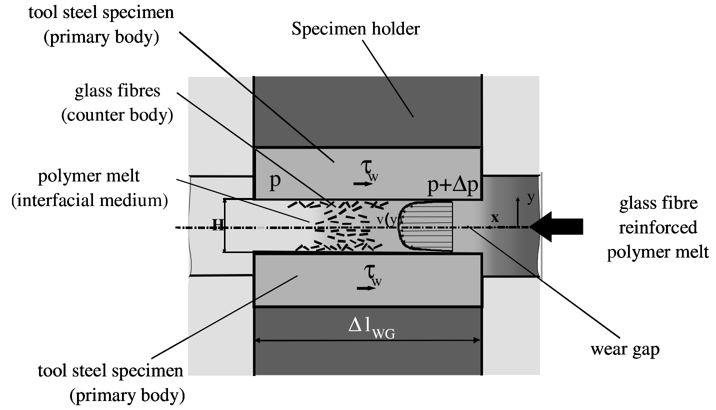


Figure 1: Tribological system „abrasive polymer melt/steel specimen“ in the wear testing apparatus with integrated measuring technology

According to DIN 50320, wear is defined as “the process of a progressive loss of material from the surface of a solid body as a result of a tribological load.” From this it follows that wear is the property of a complex tribological system [1, 3]. The elements of this system (fig. 1) in the wear testing apparatus (fig. 2) are the primary body (e.g., metallic surface of the tool steel specimen), the counter body (e.g. abrasive glass fibers in the polymer melt), an interfacial medium (polymer melt) and if existing a surrounding medium. The wear occurs during the action of the collective wear stresses on the elements of the tribological system and manifests itself in energetic and material interactions between these elements. The pressure flow in the wear gap causes the wall shear stress τ_W according to the pressure drop Δp. The non-newtonian velocity profile v(y) leads to fiber orientation in flow direction and fiber rotation near to the steel surface.

In polymer processing the main wear mechanisms adhesion (metallic contact between the screw and the cylinder) abrasion (impact from hard fillers [4] and plowing process in flowing direction) and corrosion (chemical attack from the polymer melt itself or from the reaction products from the melt and/or the additive) can occur [1, 2, 3]. In the machine components filled with polymer melt, wear is mostly caused by a superimposition of abrasion and corrosion [1, 5, 7]. These two mechanisms often interact. On the one hand surfaces are first attacked by corrosion and then abraded and on the other, passive layers are mechanically abraded and the material beneath corrodes. Degradation products of both the polymer melt and its additives react with traces of moisture to form corrosive acids which are responsible for the superimposed corrosion [5, 7]. Acids such as hydrochloric acid when processing polyvinylchloride (PVC), nitrous acid (polyamide PA), sulphuric acid (polyether sulfone PES) and phosphoric acid in case of phosphorous-containing flame retardants can be formed. In injection molding the steel surface activated by abrasion is particularly chemically attacked whenever no melt flow takes place.

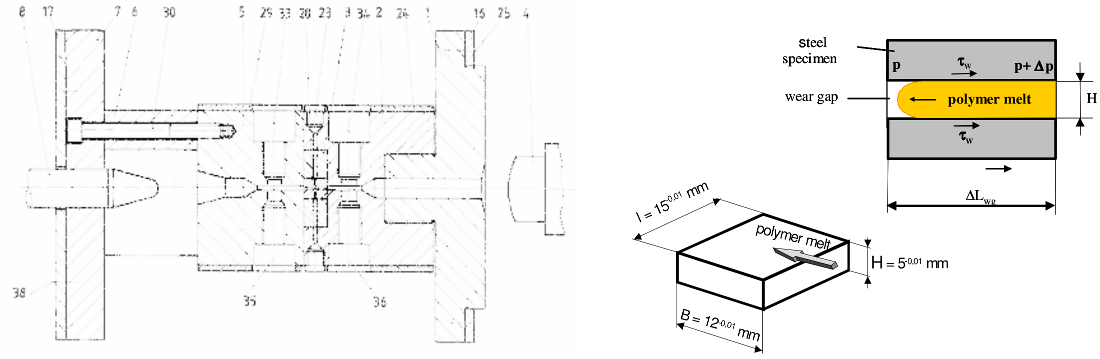
Corrosion (DIN EN ISO 8044) changes the material by chemical, electrochemical or physical means. Chemical reactions are necessary to produce corrosive products. The corrosion system consists of the material and the corrosive medium, which is made up of the chemical substances near to the exposed surface [1]. Wear (DIN 50320) changes the surface only by mechanical action. The chemical composition of worn out material particles is not changed in the process [1]. Most metals and their alloys are thermodynamically unstable against chemically reactive components like water, air (oxygen, carbon dioxide), chloric gases, acidic rests, electrolyte solutions, halogenic hydrogens, and contamination and degradation products. These components enter the polymer processing machine via the environment, additives, fillers and contaminations (degradation while melting) and the polymer melt itself (separation, degradation and cross linking). Corrosive polymers are polyamide (PA), polyvinylchloride (PVC), polyacetal (POM), acrylonitrile-butadiene-styrene copolymer (ABS), polyphenylene sulphide (PPS) and all polymers with fillers and additives which act corrosively [4].

If looked at from a purely mechanical point of view, micro plowing from fillers and reinforcements in the polymers is decisive for abrasion. The amount of material removed is essentially a function of type, amount, composition and hardness of the additives, the number of micro cutting edges and the forces, which these materials can exert, via the melt, on the steel surface. The latter is a question of a particle's own motion and kinetic energy, its fixing in a certain position by the highly viscous polymer melt, and is influenced greatly by the processing parameters (temperature and viscosity of the polymer melt, pressure and shear rate as well as filler orientation). Polyamide 66 (PA66), followed by polybutylene terephthalate (PBTP) and polyacetal (POM) are some of the most wear intensive plastics [1]. Glass fibers are by far the additive with the highest wear intensity, followed by natural silica, metal oxides, natural calcium carbonates, metal powders, flame retardants, talc, etc.

Experimental

Wear testing apparatus for the measurement of abrasive wear

Usually model wear test methods [1, 2, 3, 5] are preferred for development and they use specimens with a simple geometry (e.g. figure 2b). Wear is determined as the loss of weight related to the surface area (e.g. given in mg/cm^2) or as the depth of material removed (given in µm). Significant results are obtained after just a few material throughputs of, for example, 10 or 20 kg of plastic material. The used wear testing device (fig. 2) contains an integrated measuring technology.



(a) **(b)**

Figure 2: (a) Wear Testing Apparatus with integrated measuring technology,(b) geometry of the wear specimens
28 platelet-shaped test specimen (b); 3 specimen holder; 23 Fe/Co-thermocouple; 33, 34 pressure transducer;
35, 36 Infrared-(temperature) sensor; 1, 2 die plate; 4 machine nozzle;, Rz = 2 µm, Ra = 0.2 to 0.3 µm; direction of grinding = flow direction of the melt [3]

All of the wear relevant loads such as actual wall shear stress, wall shear rate, melt temperature and specimen temperature are measured with integrated sensors. The apparatus is built into the injection molding machine as an injection mold and fixed to the clamping plates of the machine by both flanges (position 1, 7). The wear specimens (28) in the specimen adapter (3) can be replaced quickly and easily when the clamping unit is open. They form a rectangular gap in which high local shear stresses and shear rates up to 200,000 s^{-1} can be produced. The melt is injected through the wear gap and causes the material abrasion on the surface of both wear specimens. The melt pressure (33, 34), the melt temperature (35, 36) are measured in front of and behind the wear gap. In addition the specimen temperature at different depths (1 and 4 mm beneath the wear surface) is measured. From these values and the known heat conductivity of the steel specimens the steel temperature at the wear surface during the test cycle can be calculated.

Taking into consideration the rise in melt temperature of the hot polymer melt in the non-isothermal flow through the wear gap, a method was developed by which the real wall shear stress (τ_w) in the wear gap is calculated from the measured pressure [3, 5]. Assuming an energetic wear model [3, 5], wear depends on the abrasion work per surface unit $A_{W,spec}$ (equation 1), which is a characteristic value for the abrasion process caused by the glass fiber reinforced plastic melt during flow. It contains not only the frictional energy but also the work share for melt shearing and pumping.

$$A_{W,spec.} = \tau_W^{\,m+1} \cdot \frac{\Phi}{m+2} \cdot \left(\frac{H}{4}\right) \cdot t_{tct} \tag{1}$$

It is a function of the wall shear stress in the wear gap τ_W, the viscosity of the plastics melt (material constants Φ and m of the power law formula), the wear gap H, and the total contact time t_{tct} of the polymer melt with the wear surface, which corresponds to the total injection time over all shots (total time of wear load).

$$\Delta h = \frac{m_{St}}{\rho_{St} \cdot B \cdot L_{wg}} \tag{2}$$

The removal of material (abrasion) by the abrasive glass fibers is calculated as the mean depth of abrasion Δh (eq. 2). It depends on the weight loss of the steel specimen measured (m_{st}), the density of the steel specimen (ρ_{St}) and the wear surface.

For the wear tests an injection moulding machine of the type BA 500/200 from Battenfeld Kunststoffmaschinen Ges.m.b.H., Kottingbrunn, Austria, was used. The entire shot volume of the plasticized melt was injected at a defined pressure, screw forward velocity and melt temperature (e.g., 300 °C for PA66 and 390 °C for PES). After a predetermined amount of plasticized melt had been injected (e.g., 2.5 kg, 5 kg, 10 kg, 15 kg, or 20 kg), both specimens were removed, carefully cleaned in a solvent and afterwards weighed on an analytical balance with a precision of +/- 0.05 mg. From the original weight and the weight after injection, the wear was determined as the mean depth of abrasion Δh with an error in measurement of ± 0.06 µm. Afterwards the specimens were replaced and the wear test was continued. To inject 5 kg PA66, 32 shots were necessary. Because of the high dissipation due to shearing during injection, the melt temperature rises by 20 °C to 40 °C when polyamide 66, reinforced with 50 wt.% glass fibers, is injected through a wear gap of 0.5 mm at shear rates of appr. 200,000 s^{-1} [5] The maximum shear dissipation occurs at the wear surface; therefore the surface temperature of the specimen rises from 300 °C to about 384 °C.

Corrosion testing apparatus

The new developed corrosion-testing apparatus (fig. 3) enables laboratory tests to be carried out under conditions of increased corrosion with direct contact of steel surface and polymer melts. High shear rates and high temperatures are used to accelerate the degradation of the polymer and its additives. The polymer melt is sheared in a manner similar to the steady state laminar flow in a rotational rheometer. The test cavity, filled with polymer melt, is shaped like a circular disc with a height, which varies between 0.25 mm and 2 mm. The melt temperature used is between 180 °C and 300 °C, and rotational speeds up to 540 rpm and shear rates up to 6,800 s^{-1} are possible. Measuring times are between several hours and several days. During measurement, the metal specimen is in close contact with the hot polymer melt.

The corrosion-testing apparatus (fig. 3, left) can be installed into a horizontal injection molding machine or a vertical press. The measuring cell (fig. 3, right) is clamped and heated by the mold plates (2, 3) and consists of the metal specimen (4) and the rotor head (5) (hot circle). The light blue components (6) serve to keep the bearing area at the correct temperature. The flow of heat from the hot area into the surrounding areas is reduced by polyimide plates and sleeves (7, light grey). The mold mounting plates marked in brown are not heated (8). The rotor (9) is also heated and is driven by a reduction gear unit and a direct current motor. Rotational speeds of up to 540 rpm are controlled using closed loop control and the torque adjusted as necessary. The polymer can be inserted as a pre-injected circular disk or as pellets and is melted by heat and mechanical shear. Rotational speed, torque and specimen temperature are measured during the test cycles [7].

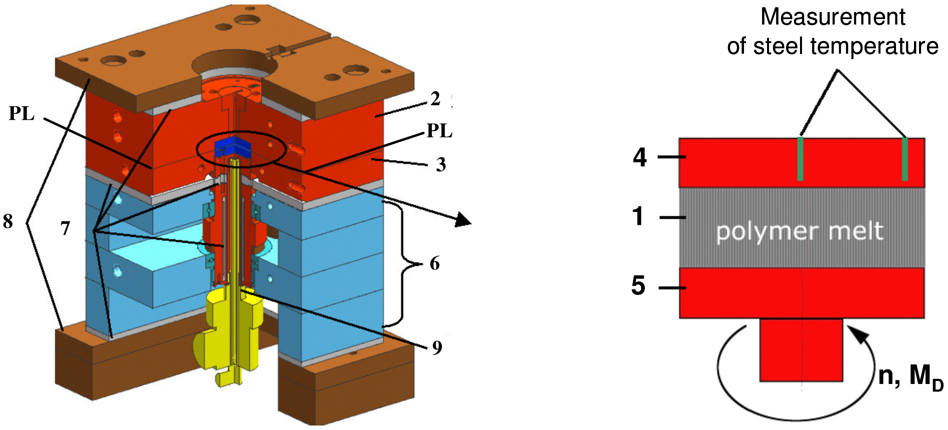


Figure 3: left, partial view of the new developed corrosion-testing apparatus [7]; right: measurement cell;
1 polymer melt, 2 upper mold plate, 3 lower mold plate, 4 metal specimen, 5 rotor head, 6 low temperature area,
7 insulating polyimide plates and sleeves, 8 mold mounting plates, 9 rotor; PL parting line

For the serial experiments plasticized polyvinylchloride at 210 °C melt temperature was used with mirror-finished test specimens. A defined order of 12 subsequent dynamic (1.25 hours) and static (1.25 hours) cycles was performed with an overall testing time of 30 hours (12 single tests in all). The temperature in the hot area of the apparatus was adjusted to 235 °C. For each single test, a mass of 3.5 g was tested and removed afterwards. In the measuring cell, a shear rate of 150 s^{-1} was applied at the outer specimen-diameter. The specimen cleaning was carried out in an ultrasonic bath. The ratio of volume change to exposed surface of the specimen was defined as a criterion for measuring the corrosion resistance.

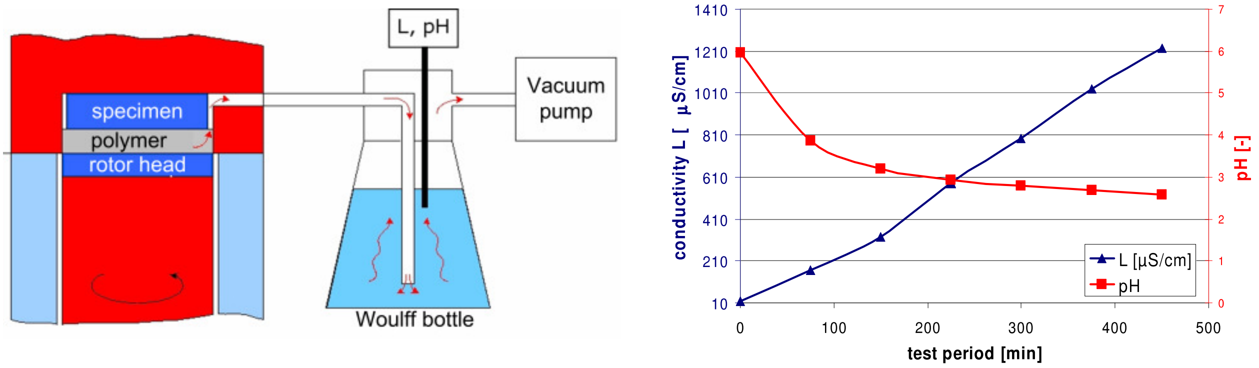


Figure 4: left, experimental set-up to measure corrosiveness; right, measurement of electrical conductivity and pH-value as a function of time

The corrosiveness of this test had been proven using electrical conductivity and pH-value measurements (fig. 4). The gaseous corrosive products (hydrochloric acid) flowed out of the corrosion testing apparatus through a small hole near the measurement cell and through a borehole. Then they the corrosive gas was dissolved in twice-deionized water. Strong changes in pH-value and electrical conductivity were measured under combined static/dynamic conditions. After 200 minutes testing time a pH-value of 3.0 was achieved which is almost similar to pH-values of the salt spray test acc. to DIN 50021 (pH from 3.1 to 3.3).

Plastic mold steels investigated

Table 1 shows the chemical composition and the heat treatment of the steel grades investigated. The steel grades M390, CPM420 V, Elmax, Fe 64 K and Supracor were produced by powder metallurgy (so-called PM-steels).

As plastic material for testing the steel grades 1.2312 and 1.2316 polyamide 66, containing 50 wt.% glass fibres, BASF-grade Ultramid A3WG 10 (abbr.: PA66+50wt.% GF) was used. The PM-steel grades M390, CPM420 V, Elmax, Fe 64

K and Supracor were tested using polyamide 66, containing 35 wt.% glass fibres, BASF-grade Ultramid A3WG 7 (abbr.: PA66+35wt.%GF). Because of the high tendency to moisture absorption, both materials were dried in a drying air drier according to the material specification with maximum relative humidity of 0.2%.

Table 1: Chemical composition and EN/DIN designation of the steel grades investigated

Steel grade	**chemical composition (EN/DIN)**							**heat treatement**	**test**
	C	Si	Mn	Cr	Mo	V	Others		
M200 1.2312	0.40	0.40	1.50	1.90	0.20	-	S 0.08	hardening: 850 °C in vacuum, tempering: 650 °C, hardness: 30 HRC	Abrasive wear
M300 1.2316	0.38	0.40	0.65	16.0	1.00	-	Ni 0.80	hardening: 1020 °C in vacuum, tempering: 650 °C hardness: 30 HRC	Abrasive wear
M333 Bohler	0.28	0.30	0.3	13.5	-	-	N 0.10	hardening: 1020 °C in vacuum, tempering: 250 °C hardness: 52 HRC	Corrosive wear
M390 Bohler	1.90	0.70	0.30	20.0	1.00	4.00	W 0.60	hardening: 1150 °C in vacuum, tempering: 250 °C tempering 525 °C, hardness: 63.1 HRC	Corrosive wear Abrasive wear
CPM420 V Crucible(Zapp)	2.2	0.4	0.5	13	1.0	9.0		hardening: 1150 °C in vacuum, tempering: 500 °C, hardness: 63.1 HRC	Abrasive wear
Elmax Uddeholm	1.7	0.4	0.3	17	1.0	3.0		hardening: 1150 °C in vacuum, tempering: 525 °C, hardness: 62.7 HRC	Abrasive wear
Fe 64 K IMT	2.6	0.5	0.4	26	1.1	2.7	Nb 1.5	hardening:1150 °C in vacuum, tempering: 525 °C, hardness: 63.0 HRC	Abrasive wear
Supracor Crucible(Zapp)	3.75	0.9	0.51	24	3.0	9.0		hardening: 1175 °C in vacuum, tempering: 570 °C, hardness: 62.5 HRC	Abrasive wear

Results and Discussion

Abrasive wear

Figure 5 shows the abrasive wear for low alloyed steel 1.2312 in comparison to the higher alloyed steel 1.2316. Each steel grade was tested with a total of 50 kg of PA66+50wt.%GF. From the data gathered the mean depth of abrasion Δh in µm and the abrasion work per surface unit $A_{W,spec}$ was calculated. Taking into account the widening of the wear gap with increasing testing time as well as the corresponding change in shear stress, shear rate and viscosity for the calculation of $A_{W,spec}$, the measured values for the low wear resistant steel 1.2312 are strongly non-linear. The highly alloyed steel 1.2316 shows a linear wear curve and Δh-values more than 10 times lower than for 1.2312 after injecting 50 kg PA66+50wt.%GF.

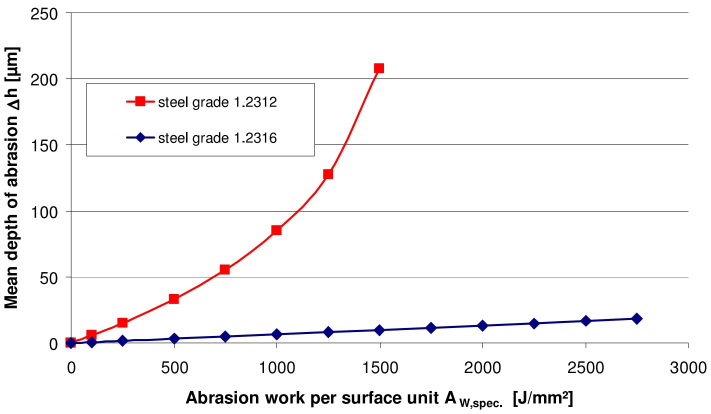
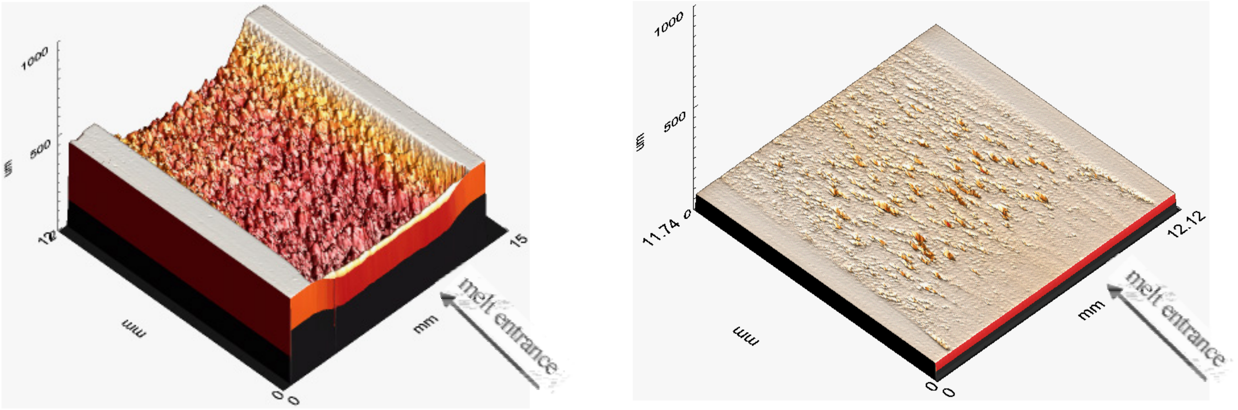


Figure 5: Mean depth of abrasion as a function of the abrasion work per surface unit for steel grades 1.2312 and 1.2316; injected plastic material in all 50 kg PA66+50wt.%GF

Figure 6 shows the 3D-topography measurements of the steel specimens, investigated fig. 5. The measurements had been carried out after injecting 20 and 25 kg PA66+50wt.%GF respectively. Despite of the great difference in abrasion due to wear resistance both pictures show a strong influence of the melt entrance geometry on wear. Using such 3D-data, wear can be calculated as volume loss with an error in measurement of ± 2.5 % for low alloyed mold steels and ± 2.5 % for highly alloyed mold steels. In addition wear profiles as a function of the distance from melt entrance can be calculated. Thus the influence of entrance geometry and pressure on wear can be investigated.



1.2312, 20 kg PA66+50wt.% GF

1.2316, 25 kg PA66+50wt.% GF

Figure 6: 3D-topography for steel grades 1.2312 (left) and 1.2316 (right), measured after injecting 20 and 25 kg PA66+50wt.%GF respectively

Figure 7 shows the abrasive wear for the highly wear and corrosion resistant steel grade M390, which was produced by powder metallurgy. In all 45 kg of PA66+35wt.%GF were injected. The mean depth of abrasion is in the range of 0.35 µm. The measured wear curve for M390 as well as for the other PM-steels can well be linearly approximated.

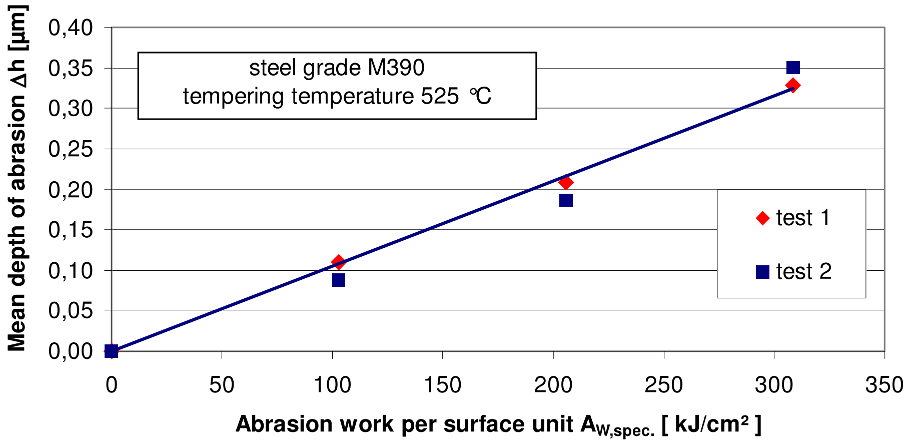


Figure 7: Mean depth of abrasion as a function of the abrasion work per surface unit for steel grade M390; injected plastic material in all 45 kg PA66+35wt. % GF (15 kg for each test cycle) [9].

To investigate the correlation between chemical composition of PM-steels and their wear resistance the thermodynamic calculation programm “Thermo-Calc” [8,10], which is a common tool in steel development, can well be used. This software allows to calculate the matrix composition, type and amount of hard phases as well as calculation of phase diagrams [8,9,10]. Wear resistance depends on type, amount and size of hard phases in the steel matrix, while the chemical composition of the matrix decides on corrosion resistance. The total amount of alloying components is shared into matrix- and hard phase-content, wheras the percentage of content is influenced by the heat treatment. As wear primarily depends on type and content of hard phases, a so-called “carbide index CI” (equ. 3) can be calculated out of hardness and hard phase content of occuring carbides [9]. Vanadiumcarbide (MC) shows a hardness of about 2,800 HV (Vickers hardness) to 3,400 HV, while chromiumcarbide (M7C3) has a hardness of about 1,500 HV [9]. These 2 carbide types are mainly part of the hard phase of PM-steels. Equation 3 shows the formula to calculate the Carbide Index CI.

$$CI = X_{M7C3} \cdot H_{M7C3} + X_{MC} \cdot H_{MC} \qquad (3)$$

It is a function of the amount X of carbide in vol.% and its corresponding hardness H in Vickers hardness HV.

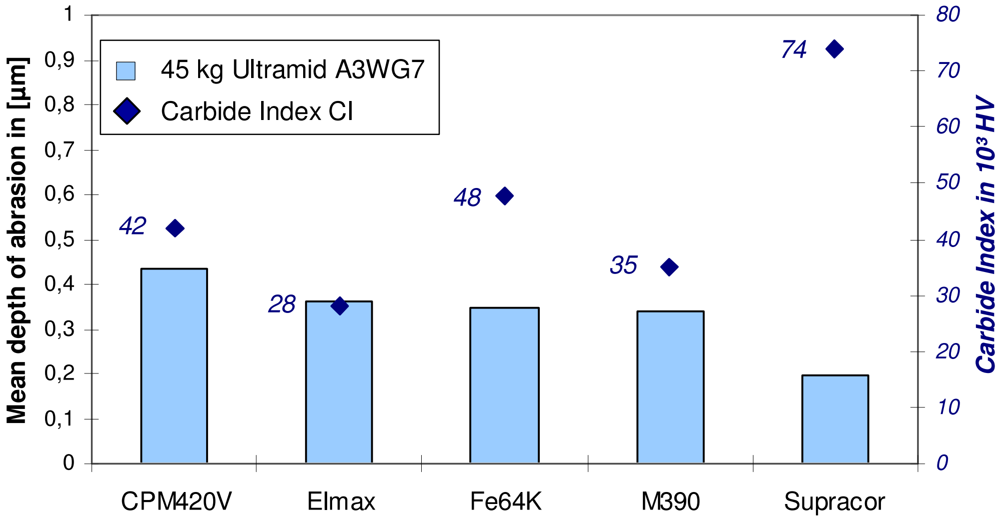
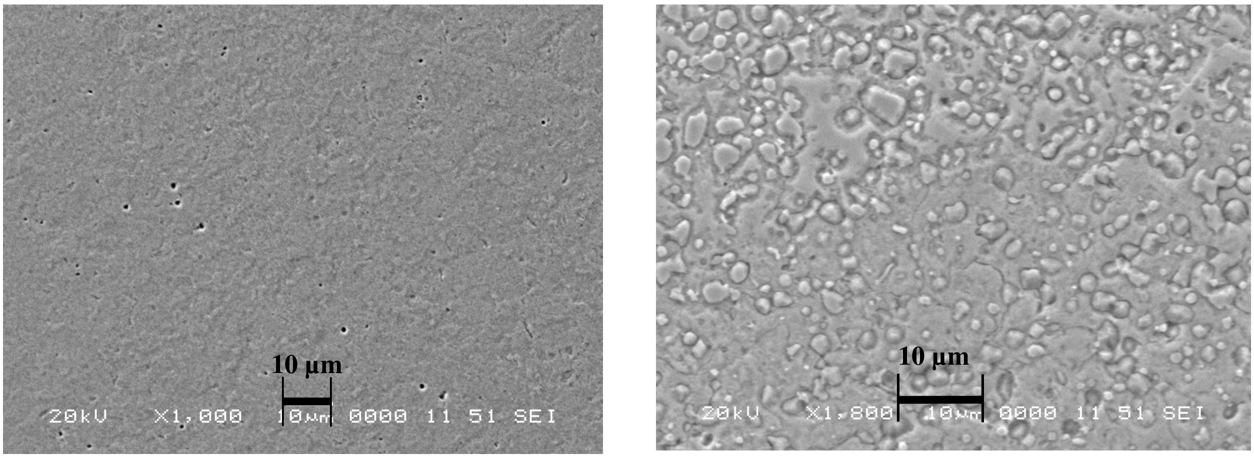


Figure 8: Mean depth of abrasion of 5 PM-steels in comparison with Carbide Indices, calculated using Thermo-Calc [9]; injected plastic material in all 45 kg PA66+35wt. % GF.

The calculated carbide indices using Thermo-Calc correspond very well with CIs, resulting via image analysis from photomicrographs of polished steel specimens [9]. Fig. 8 shows that the Carbide indices correlate well with wear resistance, expressed by the measured depth of abrasion. The best wear resistance is shown by steel Supracor, followed by M390, Fe64K, Elmax und CPM420V. Supracor has a very low toughness due to the high carbide content and is very different to machine while M390 Microclean exhibits a much better toughness and machinability [10]. Nowadays the PM-steel Supracor is offered as bi-metallic HIP clad component (so-called CRU-CLAD) in combination with a tough base material twin screw blanks and twin screw barrels [11].

Corrosive wear

The corrosion-induced volume change of the steel specimen is determined by using a non-contact, optical 3D-topography measuring instrument FRT MicroProf®. The corrosion phenomena like uniform corrosion, local pitting, corrosion of grain boundaries and corrosion of carbide-matrix boundaries are analyzed using an optical microscope and scanning electron microscopy SEM-EDX.



Bohler M333 Isoplast, 250 °C tempering temperature

Bohler M390, 250 °C tempering temperature

Figure 9: SEM pictures of steel grade Bohler M333 Isoplast (left) and Bohler M390 Microclean (right).

The Bohler steel grade M333 Isoplast with a mirror-finished surface and the Bohler steel grade M390 Microclean, tempered at 250 °C, showed the highest corrosion resistance (fig. 9). Nevertheless local pitting and slight corrosion of grain boundaries (fig 9, left) as well as slight corrosion of grain boundaries and local corrosion at carbide-matrix boundaries (fig 9, right) had been detected.

Conclusion

The wear testing apparatus with integrated measuring technology allows the determination of the abrasive/corrosive wear on steels for plastics moulds quickly and with good reproducibility. Wear is plotted as the mean depth of abrasion (error in measurement of ± 0.06 µm) dependent on the abrasion work per surface unit. Wear can either be dedected as mass loss by weight measurement or as volume loss using a non-contact 3D-topography measuring instrument. The temperature on the wear surface of the steel specimen is up to about 80 °C higher than the temperature of the melt due to shear dissipation. For wear resistant steel grades like highly alloyed steels produced via electro slag remelting and PM-steels linear wear curves were determined. Increasing hardness as well as increasing surface quality (high gloss polishing) leads to lower wear. The highly alloyed PM-steel Supracor containing 3.75 % C, 24 % Cr und 9 % V showed the best wear resistance, followed by M390 Microclean, which however exhibits a much better toughness and machinability. The abrasive wear very well corresponds with a so-called "carbide-index", calculated from the volume content and hardness of chromium- and vanadium-carbides.

The corrosion-induced wear of different plastic mold steels was investigated using a new practical apparatus, which is designed like an injection mold and works similar to a plate/plate rheometer. The heaviest corrosion attack was achieved applying a sequence of 12 single tests with 180 min testing time for each single test under alternating dynamic and static conditions. As corrosive polymer plasticized polyvinylchloride was used. The corrosion attack was induced by mechanical and/or thermal degradation producing hydrochloric acid. After 200 minutes testing time a pH-value of 3.0 was achieved. The corrosion-induced wear of the metal specimens was examined by measuring their change in volume using a non contact 3D-topography measuring instrument. The corrosion phenomena like uniform corrosion, local pitting, corrosion of grain boundaries and corrosion of carbide-matrix boundaries were analyzed using an optical microscope and scanning electron microscopy SEM-EDX. The Bohler steel grade M333 Isoplast with a mirror-finished surface and the steel grade M390 Microclean, both tempered at 250 °C, showed the highest corrosion resistance, nevertheless local pitting, corrosion at grain boundaries and local corrosion at carbide-matrix boundaries had been detected.

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