



# Effect of heat treatment, lubricant and sintering temperature on dry sliding wear behavior of medium alloyed chromium PM steels

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## ABSTRACT

The influence of sintering temperature, heat treatment and lubricant on the wear rate of warm compacted Fe–3%Cr–0.5%Mo–0.45%C Astalloy steel P/M alloy [Astalloy CrM] was investigated using dry sliding wear test, X-ray diffraction and metallographic examinations. Observations showed that for warm compacted Astalloy CrM heat treatment by quenching and tempering at 450 °C reduces wear rate, while tempering at lower temperatures such as 200 °C has the adverse effect. In this case dominant mechanism was found to be delamination wear as evidenced by lustrous large metallic debris. Two different lubricants, i.e. Li stearate and stearamid were investigated. For specimens sintered at high temperature, Li stearate lubricant shows better wear behavior, whereas for low temperature sintering, stearamid will be a better lubricant regarding wear behavior of sintered product. Measurements of transverse rupture strength and wear rate confirmed that pores play a major role in both static and dynamic properties of PM parts.

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## 1. Introduction

Recent developments such as warm compaction contribute to creating PM parts with high sintered density and high mechanical properties and hence, have found wide application in the industry. One of the main areas for PM parts is wear application and sliding parts like gears, sprockets, cam lobes and similar parts which are mostly used in automotive and office machine industries. Hence, the wear behavior of warm compaction PM parts was investigated in this research.

Wear behavior of wrought steels has been the subject of intense studies, and a fair amount of data has been collected for various types of steels under different conditions of ear-

ring environments. For these materials, attempts have been made to explain the wear behavior of materials through their intrinsic properties, and some well-established relations exist.

However, one expects considerable scatter of data, mainly due to the nature of the test and the numerous parameters involved (Guicciardi et al., 2002), as well as the unwanted heat treatment effects of the contact surfaces (Scherge et al., 2003).

An important parameter in wear rate is the sliding speed. Lee (2004) has shown that the state of strain on the worn surfaces has a good relationship with the state of wear. He has also shown that the magnitude of strain on worn surfaces corresponds to the wear rate, and the increasing rates of strain with sliding distance presumably correspond to the wear

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modes. However, Wang and Danninger (1998) have found that maximum wear rate for sintered Fe–3.5%Mo–1% C material varies with both sliding speed and contact load, the transitional speed from mild to severe wear decreasing from around  $8 \text{ ms}^{-1}$  for contact load of 10N to around  $2 \text{ ms}^{-1}$  for 60N. According to Lim (2002), the trend of the wear rate is also predictable from these maps. The wear map depends however on the counter material and the environment, especially the atmosphere.

However, the data for PM steels is rather scarce, and is not as well documented as for wrought materials. There are also additional complications due to the presence of pores and other metallurgical features peculiar to sintered materials (Simchi and Danninger, 2004; Wang and Danninger, 2001). The presence of pores may have a positive effect by reducing the wear rate, since the pores can act as reservoirs for oil in wet sliding (Simchi and Danninger, 2004), or as traps for debris in dry sliding (Lim and Brunton, 1986). In this case however, the porous material can approach at best the behavior of fully dense one. Decreasing the wear rate as a result of trapping of the debris in the pores in a PM part has been attributed to: (1) decreased contact pressure as compared to porous material without debris entrapped in pores, i.e. with fully open pores, (2) reduced possibility of formation of large abrasive particle agglomerates during sliding and (3) decreased possibility of plastic deformation near the pores and formation of less metallic debris (Dubrujeaudet et al., 1994). However, it is important to note that this behavior of porous PM material is quite sensitive to the level of porosity.

Danninger et al. (2003) and Wang and Danninger (2001) have shown that for Fe–1.5%Mo–0.7%C PM alloys in both as sintered and heat-treated conditions, dry sliding wear of material with more than 10–12% porosity is nearly twice that of material with less than 5% porosity. The authors have attributed this jump to transition from closed to open pores. Simchi and Danninger (2004) have suggested that the optimum level of porosity for least amount dry wear in plain iron PM material [ASC 100.29-Höganäs composition 0.01% C, 0.07% O] is around 10%.

Another important parameter is the effect of heat build up as a result of severe contact between pin and the disk. This localized flash heating can cause tempering effects and as a consequence, change of microstructure and hence, the wear rate. Temperature rise as much as  $1000^\circ\text{C}$  has been reported (So et al., 2002). Debris formed during the sliding process vary in shape, size and composition depending on the wear mechanism(s) involved. They can vary from coarse metallic particles to very fine oxides of iron [hematite and magnetite] in the nanometer range. Once the first debris are formed, these particles will separate the sliding surfaces and change the tribological conditions. Therefore, wear rate will now depend on the friction coefficient of these materials and can transform from that of mild-oxidation to severe-oxidation wear. This effect has been studied by Kato (2003) by measuring the wear rate after intentional addition of oxide particles of different size to the interface between rubbing surfaces.

However, it should be pointed out that one does not expect similar behavior for indigenous oxides. There have been contradictory findings as regards to the effect of heat treatment on PM steels.

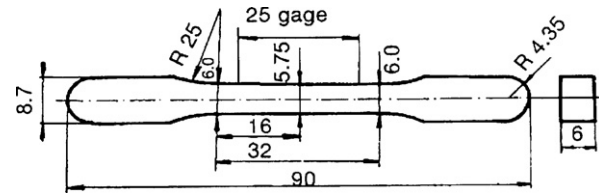


Fig. 1 – Shape of tensile test bar used for turning and preparing wear test.

Khorsand et al. (2002) have observed that the wear rate of Fe–1.75%Ni–1.5%Cu–0.5%Mo–0.6%C PM material in as-sintered condition is about twice as much as that of the same material after heat treatment (austenitization at  $800\text{--}850^\circ\text{C}$  plus oil quench and tempering to  $300\text{--}350^\circ\text{C}$  for 60 min). Haseeb et al. (2000) have reported doubling of wear rate of ductile iron wrought materials when going from austempered state to quench and tempered condition at the same level of hardness (455 HV). Some researchers have reported higher wear rates for heat-treated materials as compared to the as-sintered specimens (Wang and Danninger, 1998).

These uncertainties in prediction of wear properties of PM materials of different nature necessitate the exclusive evaluation of wear behavior of each material made from a specific powder and by a different production route.

## 2. Experimental details

The steel powder Astaloy CrM which is a water atomized pre-alloyed powder (Fe–3%Cr–0.5%Mo) with a particle size under  $150 \mu\text{m}$  and 0.45% C (by addition of natural graphite) was used for this experiment. The test samples were prepared from two types of powders: the first one admixed with 0.6% lithium stearate, and the second with 0.6% stearamid as lubricant. The powder mixture was warm compacted at  $150^\circ\text{C}$  in a pressing tool with floating die for flat tensile test bars according to ASTM Standard E8 (Fig. 1) under compacting pressure of 600 MPa.

The samples were dewaxed at  $600^\circ\text{C}$  for 60 min in flowing nitrogen and then sintered in a laboratory push type furnace (Type AHT) with gas tight superalloy retort in flowing high purity nitrogen at two temperatures of  $1120^\circ\text{C}$  and  $1250^\circ\text{C}$ , for 60 min. Part of the specimens was austenitized for 60 min at  $900^\circ\text{C}$  in the pusher furnace and then quenched in oil. After quenching, the specimens were tempered at  $450^\circ\text{C}$  and  $200^\circ\text{C}$  for 45 min in nitrogen atmosphere.

Wear test samples were made by turning one end to form a pin 5.5 mm in diameter. The circular end face of the samples was polished to a near mirror finish.

The counter disks were made of 100 Cr 6 ball bearing steel (AISI 52100) ground flat to a nearly mirror finish. The discs had a uniform hardness of about  $62 \pm 1$  HRC.

Wear tests were carried out on a pin-on-disk wear tester according to ASTM G99-95a standard. Dry sliding tests were carried out in air at a sliding speed of  $2 \text{ ms}^{-1}$  at 40N loads.

The samples were tested for at least 43,200 m sliding distance to safely eliminate run-in effects. Temperature and

**Table 1 – Designation and properties of test samples**

Designation	Sintering temperature (°C)	Heat treatment	Lubricant	HV 30	K10E–15 (m <sup>3</sup> /mN)	TRS (MPa)	$\rho$ (g/cm <sup>3</sup> )
A	1120	AS	Li stearate	269	151.4	555	7.22
B	1250	AS	Li stearate	299	88.9	713.7	7.32
C	1120	AS	Stearamid	271	134.4	597.5	7.24
D	1250	AS	Stearamid	312	112.4	823.3	7.34
A'	1120	Q+T (450 °C)	Li stearate	383	39.1	871	7.22
B'	1250	Q+T (450 °C)	Li stearate	415	17.3	1395.6	7.32
C'	1120	Q+T (450 °C)	Stearamid	378	20.9	919.5	7.24
D'	1250	Q+T (450 °C)	Stearamid	405	12.1	1805	7.34
A''	1120	Q+T (200 °C)	Li stearate	400	40.5	1088.9	7.22
B''	1250	Q+T (200 °C)	Li stearate	564	44	1427	7.32
C''	1120	Q+T (200 °C)	Stearamid	467	30.1	1181	7.24
D''	1250	Q+T (200 °C)	Stearamid	503	60.4	1884.3	7.34

AS: as sintered.

humidity of the testing environment were not controlled, but occasionally measured by a maximum–minimum digital device to ensure near constant conditions of 45–55% relative humidity at 20–23 °C.

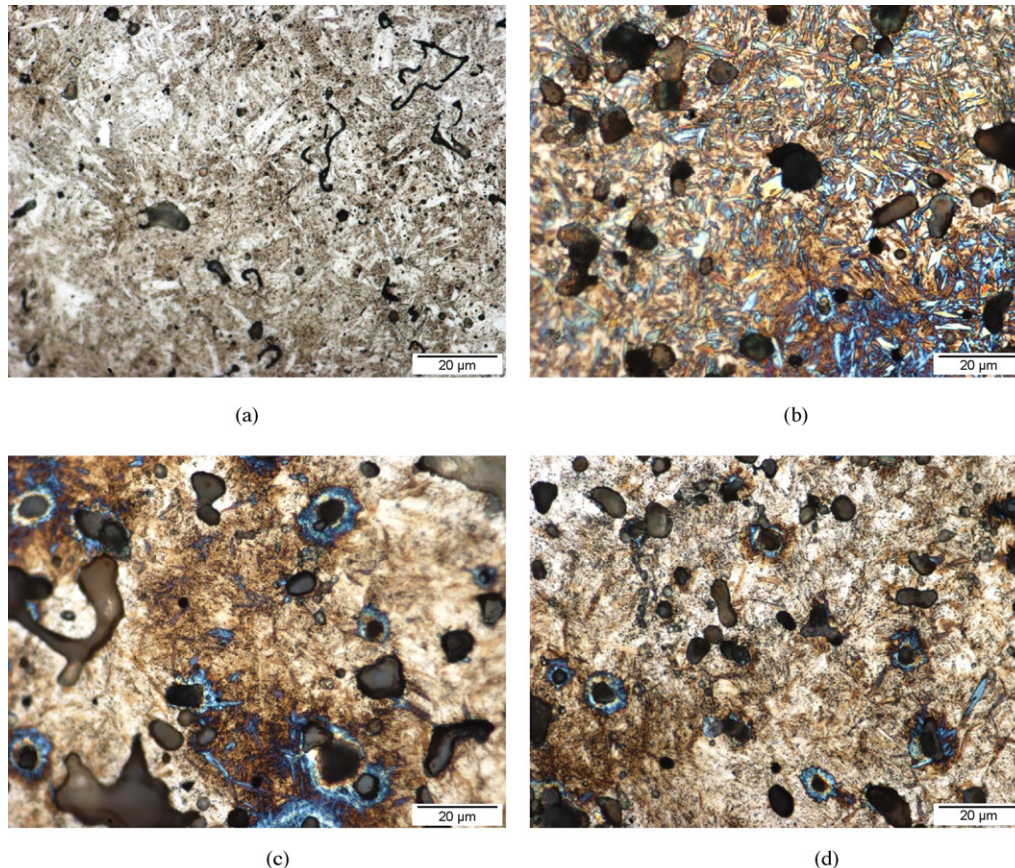
The room temperature transverse rupture strength was measured for samples in as sintered and as heat treated.

Transverse rupture strength (TRS) test were undertaken at a crushing speed of 1.0 mm min<sup>-1</sup> using a Zwick 1474 universal testing machine with a loading capacity of 100 kN. The distance between the supporting rods was 24.5 mm. The diameter of the support was 3.2 mm (according to DIN ISO 3325).

The Vickers core macrohardness was measured with a load of 30 kg on cross section, using the hardness tester (Emco Test Automatic M4U 025), from carefully sectioned and polished specimen according to DIN 51 225. The mean value of hardness for each sample was calculated by averaging a minimum of three indentations.

### 3. Results and discussion

The influence of sintering temperature, lubricant and heat treatment on the wear behavior were investigated. Designa-



**Fig. 2 – Microstructure of Astaloy CrM sintered steel. (a) Astaloy CrM AS (B') × 1000; (b) Astaloy CrM AS (B'') × 1000; (c) Astaloy CrM AS (D') × 1000; (d) Astaloy CrM AS (D'') × 1000.**

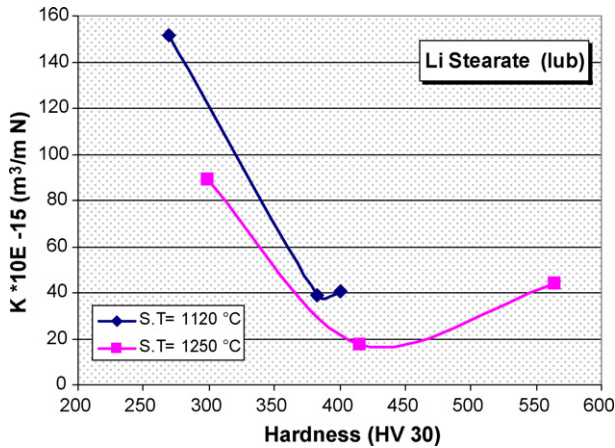


Fig. 3 – Variation of wear rate with sintering temperature and hardness (at present of Li Stearate).

tion and properties of all tested specimens are summarized in Table 1.

The effect of various parameters are as follows.

### 3.1. Microstructure

Microstructure of the investigated P/M steel alloys was found to vary with sintering and heat treatment conditions. These structures affect mechanical properties and wear behavior.

Fig. 2 shows the metallographic structure of the samples after a Nital etch. The structure consists of pearlite and bainite in as-sintered samples. Tempered martensite was found in the heat-treated samples. At low tempering temperatures, the typical fine structure of highly tempered martensite is evident.

Metallographic investigations showed that heat treatment had no effect on the shape and size of pores.

The effect of porosity on wear behavior is largely dependent on the wear condition. At different application conditions porosity shows beneficial and detrimental effects in the wear resistance of P/M steels. Microstructure contributing to the wear resistance can be ordered as follows: carbide, martensite, bainite and lamellar pearlite (Wang and Danninger, 2001).

### 3.2. The influence of sintering temperature, heat treatment and lubricant on the wear rate

To investigate the influence of the above parameters on the wear behavior, the samples were tested at  $2 \text{ m s}^{-1}$  and 40 N. The mass loss during wear process was measured and the wear rate calculated from the slope of the curves of weight loss versus sliding distance, setting aside the run-in period. In all cases, the run-in period showed to be finished within the first 1-h test run. Each sample was tested for at least 6 h runs, i.e. 43,200 m. The variation of wear with different parameters under investigation are presented in Figs. 3–6. The as-sintered specimens have higher mass loss compared to the heat-treated specimens. Heat treatment by quenching and tempering at 450 °C was found to have favorable effect on wear rate. On the other hand, tempering at 200 °C resulted in increased wear rate.

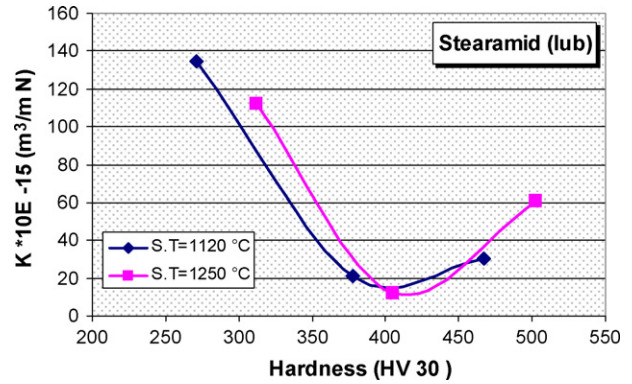


Fig. 4 – Variation of wear rate with sintering temperature and hardness (at present of Stearamid).

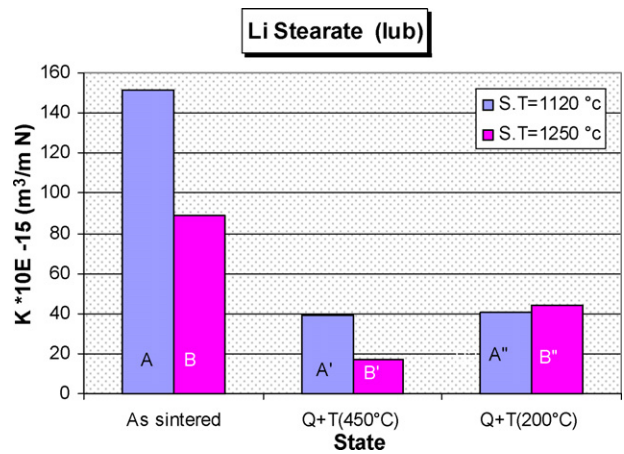


Fig. 5 – Variation of wear rate with sintering temperature and heat treatment (at present of Li Stearate).

The wear rate of the specimens sintered at 1250 °C, for both as sintered and heat treated and tempered at 450 °C, is smaller than that of specimens sintered at 1120 °C. For specimens tempered at 200 °C, those sintered at lower temperature shows a small increase in wear rate.

Tempering temperature determines the decomposition of retained austenite in the tempered martensitic and, therefore,

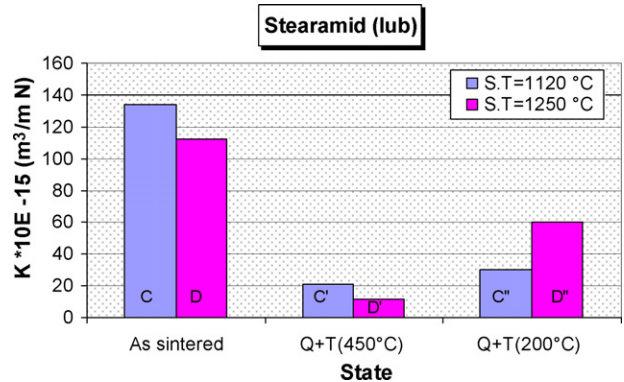


Fig. 6 – Variation of wear rate with sintering temperature and heat treatment (at present of Stearamid).

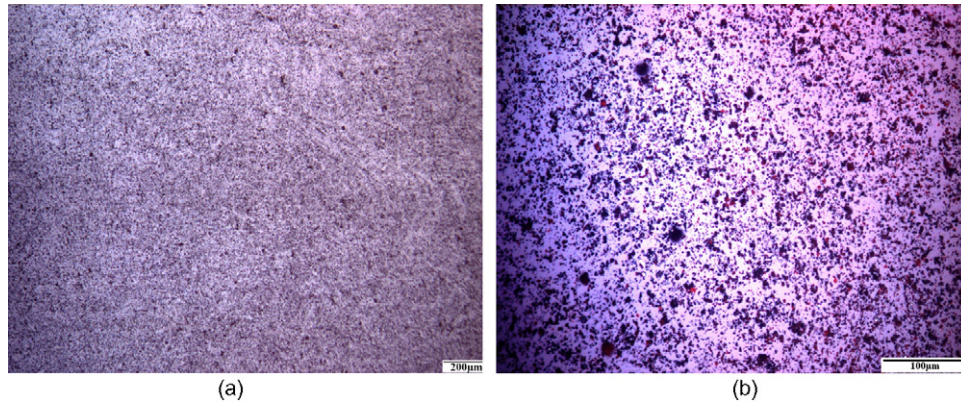


Fig. 7 – Debris formed during test. (a) Astaloy CrM AS (B')  $\times$  50 and (b) Astaloy CrM AS (D')  $\times$  200.

influences their wear behavior. At low tempering temperatures, less retained austenite is transformed to ferrite and carbides and tempered martensitic steels have high wear resistance. At high tempering temperatures, in contrast, much ferrite is transformed through decomposition of retained austenite and hardness of tempered martensitic steels decreases obviously and correspondingly the wear resistance decreases (Wang and Danninger, 2001).

Steels quenched and tempered to their peak hardness consist of martensitic matrix and carbide. The role of the carbide phase in the wear resistance of steel is also a matter of conflict. It has been reported that an increase in the volume fraction of the carbide phase enhances the wear resistance of steels. This is based on the observation that carbides are the hardest phases in steels and they have an important influence on wear resistance.

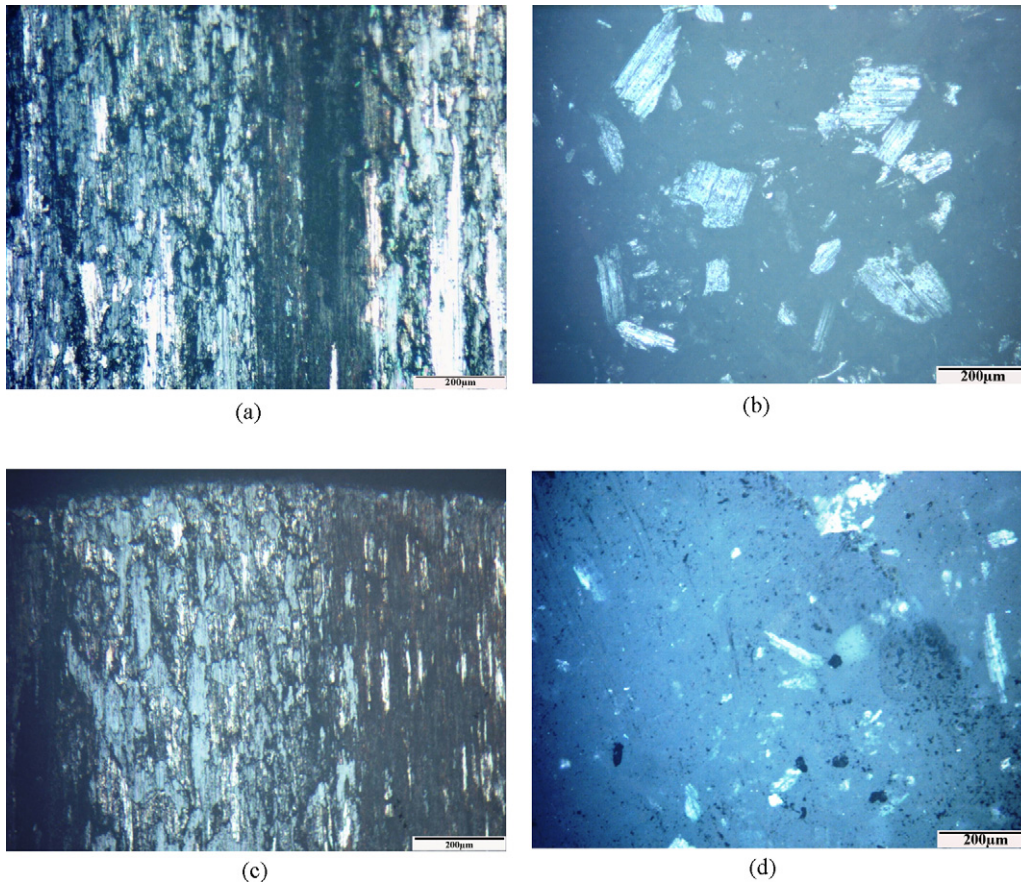


Fig. 8 – Debris and pin outer surfaces of Astaloy CrM quenched and tempered at 200 °C. (a) Astaloy CrM AS (B'')  $\times$  50 (pin outer surface at steady state); (b) Astaloy CrM AS (B'')  $\times$  50 (debris mostly rough iron particles); (c) Astaloy CrM AS (D'')  $\times$  50 (pin outer surface at steady state); (d) Astaloy CrM AS (D'')  $\times$  50 (debris mostly rough iron particles).

Hence their wear rate should be much lower than that of the matrix and, therefore, increasing the carbide volume fraction should result in a decrease in the wear rate of the steels (Clayton, 1980).

As discussed above, the wear rate cannot be simply related to heat treatment, but may correlate with its end result, hardness.

In general, it may be argued that the effect of hardness on wear rate of PM material varies with different parameters, such as porosity, heat treatment and composition.

Since composition and almost the level of porosity in the samples used in this research are the same, so except heat treatment, it is logical to assume that a parameter other than heat treatment may affect the wear behavior and wear mechanism.

The low wear rate of materials quenched and tempered at 450 °C compared to the as-sintered samples can be explained by increases in hardness and change of wear mechanism. The dominant mechanism of this species can be the oxidation wear due to oxidation of the softer constituents, as can be seen from metallographic and X-ray examination of debris.

Debris, as shown in Fig. 7, are very fine particles and X-ray examination of these particles proved it to be mixture of iron oxides and iron.

Compared to the as sintered, and quenched and tempered to 450 °C, samples tempered to 200 °C show increase in were rate. This difference can be explained by the difference in wear mechanism. The wear mechanism for this sample was found to be delamination wear as evidenced by lustrous large metallic debris (Fig. 8).

For specimens sintered at low temperature, samples using stearamid lubricant have lower wear rates compared to those with Li stearate lubricant, but for high temperature sintering, Li stearate shows better wear behavior. This can be related to the amount of porosity. Metallographic investigation shows that the amount of porosity, at low temperature sintering, for the samples that used stearamed (as lubricant) are more than samples that used Li stearate. The presence of pores may have a positive effect by reducing the wear rate, since the pores can act as traps for debris in dry sliding (Lim and Brunton, 1986). High temperature sintering decreases and improves the porosity (rounded and closed pores), and hence for high temperature sintering Li stearate has insignificant effect on improvement in wear behavior.

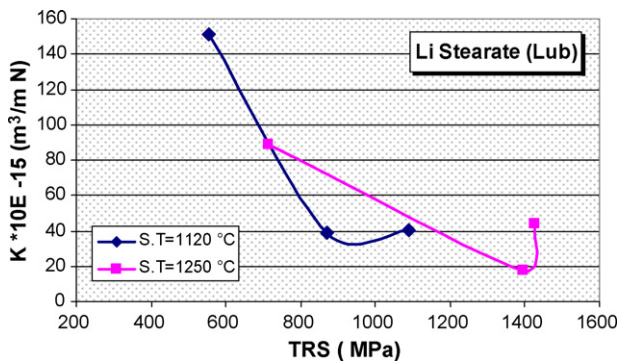


Fig. 9 – Variation of wear rate with transverse rupture strength (at present of Li Stearate).

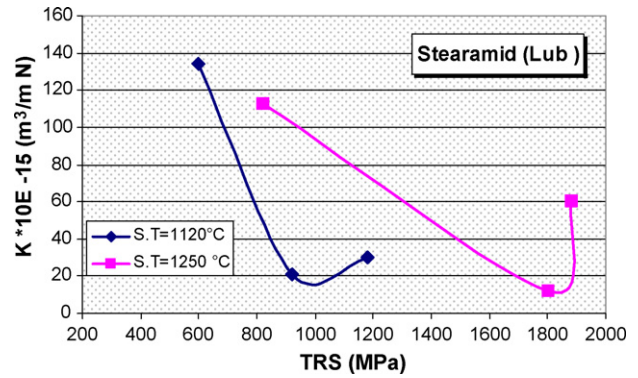


Fig. 10 – Variation of wear rate with transverse rupture strength (at present of Stearamid).

### 3.3. The influence of transverse rupture strength

Wear rate variation with transverse rupture strength for two different lubricants at two temperatures are given in Figs. 9 and 10.

The bend strength of the samples increases with increasing sintering temperature and decreasing tempering temperature. As shown in Figs. 9 and 10, the wear rate first reduces with increasing TRS of around 1400 MPa [for Li stearate] and 1800 MPa [for stearamid] and then increases with further increases in TRS (bending test results are not available for as-sintered samples).

## 4. Conclusion

Summing up the observation in this study, following points may be concluded:

- Increased sintering temperature in warm compacted Astaloy CRM PM parts, increases wear rate.
- The wear rate first reduces with increased hardness up to around 400 HV 30, due to heat treatment, and then increases with further increase in hardness. Heat treatment by quenching and tempering at 450 °C was found to have favorable effect on wear rate. On the other hand, tempering at 200 °C resulted in increases wear rate. In this case the dominant mechanism was found to be delamination wear as evidenced by lustrous large metallic debris.
- For specimens sintered at low temperature, stearamid is a more efficient lubricant as compared with Li stearate, but for high temperature sintering, samples with Li stearate lubricant show lower wear rate.
- Pores were found to play major role in TRS and wear behavior of the P/M materials. Open pores, act as sites of collection wear debris

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