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# **Evaluation of The Degradation of Combustion Engine Valves**

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This work aims at the description of the degradation of the intake and exhaust valve of the combustion engine during operation. For the experiments, new valves from Ford Fiesta were used, as well as the used ones with known mileage. In order to simulate the valve long-term operation and to estimate the temperature of the valve during the use, the new valves were annealed at 700 – 900 °C for 1000 h (corresponding to the mileage of 70,000 km with average speed of 70 km/h) and the grain size was compared with the used valves. It was found that this model experiment can easily predict the operation temperatures of the exhaust valves, which were made of the heat-resistant austenitic steel. However, the metallography procedure for the determination of the grain size fails in the case of the martensitic steel valves.

Keywords: Combustion engine, Poppet valve, Degradation, Metallography, Exhaust valve

#### 1 Introduction

Internal combustion engine (ICE) is still the most widely used power for the transport vehicles. Even though the electrification of the passenger cars is ongoing, there will be probably lots of ICE powered cars, heavy trucks, busses, ships and other vehicles still in use in the future as recent accumulators do not allow complete ICE replacement especially at large engines used for long range transport [1]. In order to keep maximal efficiency and relatively eco-friendly operation, the performance of the valves is of high importance. There are many solutions existing for the valves, such as bi-metallic valves with Stellite alloys hardfacing [2,3], valves with sodium cooling insert [4], bulk titanium valves [5] or titanium nitride coated ones [6], nickel-based superalloys, ceramics and even materials based on intermetallics are considered [7,8,9]. However, these solutions except the first one are used almost only for high-performance engines, such as sport cars, racing bikes etc. In standard engines, the current solutions are still very similar to the engine valves from 1950's, especially for intake ones. For these valves, the "Silchrome 1" martensitic steel is the most widely applied one [10]. On the other hand, the currently used materials of the exhaust valves are mostly the heat-resistant austenitic steels, while "Silchrome 1" type alloys were used in exhaust role substantially until the 1950's [11].

Even though the common material solutions for the valves have been used for many decades, there is no systematic study of their degradation during operation in the engine. There exists a series of failure analyses of defective valves [12,13] and evaluation of the wear through the analysis of the oil contamination [14,15,16]. Despite to that, the possibilities of the worsening of the mechanical properties in engines operated for high mileage due to continuous changes in the microstructure lack the adequate attention. However, such knowledge, as well as exact description of the temperature distribution in the valve, are of high importance when designing the engine and proposing a material solution for the valves. There are available results of simulation of the temperature in various parts of the valves [17], but the experimental validation is lacking. Therefore, this study aims at the description of the changes of the grain size during thermal exposure of the valves and identification of the temperatures of the used valves based on it.

## 2 Materials and Methods

The subjects of the experimental works were intake and exhaust valves from Ford Fiesta VI (2011) car with four cylinder petrol engine, in two intake as well as two exhaust valves per cylinder configuration. Both new and used ones were used. Chemical composition of the intake and exhaust valves determined on the valve head is summarized in Table 1. The chemical composition was determined by energy dispersive spectroscopy (EDS) by the means of TESCAN VEGA 3 LMU scanning electron microscope equipped with the Oxford Instruments X-max 20 mm² EDS analyser.

Chemical composition of the exhaust valves is similar to X53CrMnNiNbN21-9 standard, however containing ca. two percent silicon and one percent tungsten more, probably improving its thermal stability. Intake valves are similar to mentioned Silichrome 1 standard.

**Tab. 1** Chemical composition of the studied intake and exhaust valves (wt. %, determined by EDS)

Valve	Fe	Cr	Mn	Ni	Si	Nb	W
exhaust - new	59.8	21.4	9.1	3.7	2.8	2.3	1.0
exhaust – used	60.5	22.1	9.4	3.9	2.3	1.1	0.8
intake - new	85.4	9.7	0.3	0.4	4.2	-	-
intake - used	86.5	9.4	0.4	0.2	3.5	-	-

The mileage of the engine in the time of the exchange of the valves was 70,000 km. There was no visible damage of the valves, the reason for the repair was different. The samples of the new valves were annealed at 700, 800 and 900 °C for 1000 h. This annealing was used in order to simulate the mileage of 70,000 km with the average speed of 70 km/h (i.e. 1000 hours at the operating temperature). The annealed samples were compared with the used samples in various regions of the valve in order to determine the temperature on the valve during the operation in the engine. For the comparison, the observation of the microstructure and hardness measurement were used. The microstructure was observed after grinding on P180 -P2500 sandpapers with SiC particles, polishing by D2 diamond paste and Eposil Non Dry suspension. Etching was done by 10% nital (10 ml HNO3, 90 ml

ethanol) and by oxalic acid (20 g oxalic acid, 180 ml water, electrolytically at 6 V) for intake and exhaust valves, respectively. To reveal the grain boundaries, alternative etching by picric acid solution (1 g picric acid, 95 ml  $\rm H_2O$ , 5 ml  $\rm HCl$ ) with subsequent slight polishing was tested [18]. Microstructure was observed by Nikon MA200 optical microscope and documented by the means of NIS BR software. Grain size was determined by image analysis using ImageJ software package.

#### 3 Results and Discussion

Microstructure of the new and used intake valves is presented in Figure 1. It can be seen that the microstructure is composed of a heavily tempered martensite.

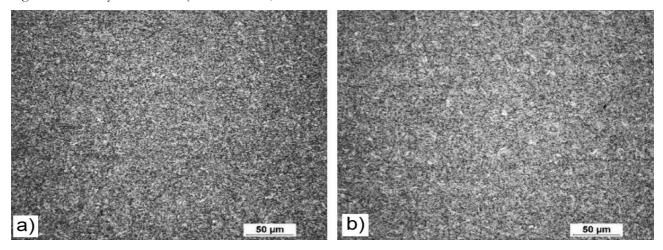


Fig. 1 Microstructure of new (a) and used (b) intake valves (etched by 10% nital)

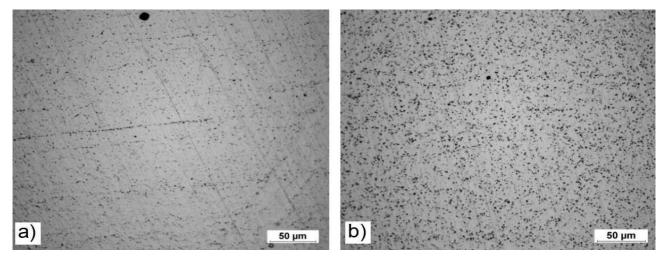


Fig. 2 Microstructure of new (a) and used (b) intake valves (etched by picric acid and subsequently polished)

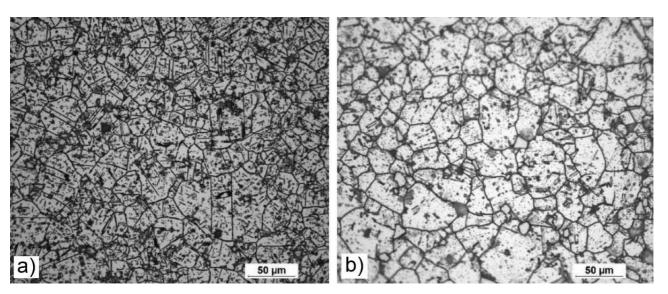


Fig. 3 Microstructure of new (a) and used (b) exhaust valves (electrolytically etched by oxalic acid)

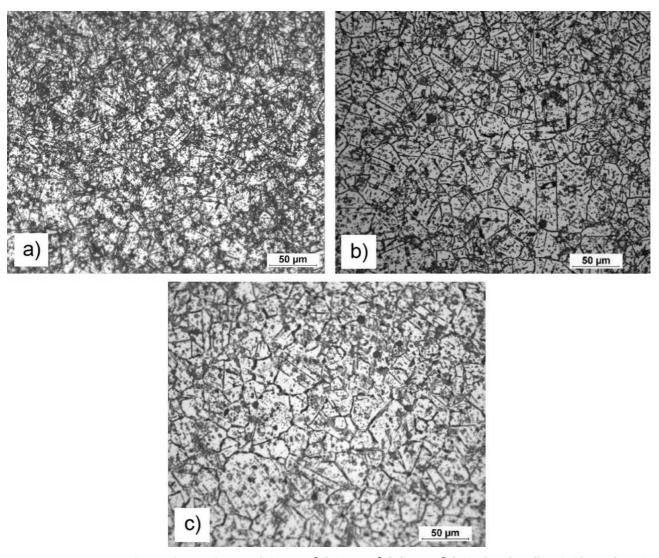


Fig. 4 Microstructure of new exhaust valve annealed at 700 °C (a), 800 °C (b), 900 °C (c), electrolytically etched by oxalic acid

Because the nital etching does not allow to reveal the grain boundaries in the case of this alloy, the procedure according to the ASTM E112-13 method was tested. The applied method is based on over-etching by picric acid solution, followed by polishing. It is expected that the whole structure is attacked by etching, while the etching of the grain boundaries is stronger. During subsequent polishing, the grain area is polished and becomes lighter on the micrograph, while the grain boundaries remain dark. However, this procedure does not work for this particular martensitic steel with present heat treatment, as can be seen in Figure 2. The grain boundaries are not visible sufficiently. Therefore, we can conclude that we are unable to use described metallographic approach in order to evaluate the degradation of the intake valves made of this martensitic steel neither in the as received heat treatment nor in the condition after the mentioned mileage.

In the case of the exhaust valve, the microstructure composed of austenitic grains with well-visible boundaries is observable, see Figure 3. Moreover, there are carbides, which contain high amount of niobium, as determined by EDS analysis, i.e. the so-called MC type.

Because the grain size was not identifiable for intake valves, the study with annealing of new valves was carried out for exhaust valves only. The microstructure of the exposed samples is shown in Figure 4 and the determined grain size vs. annealing temperature is shown in Table 2.

Tab. 2 Average grain size of annealed samples cut from new exhaust valve head

State	Grain size (μm)
New	45 ± 5
annealed at 700 °C	45 ± 7
annealed at 800 °C	47 ± 7
annealed at 900 °C	52 ± 6

The values in Table 2 were fitted by Sellars model - equation (1):

$$d - d_0 = A \cdot t \cdot e^{-\frac{Q}{RT}} \tag{1}$$

Where:

d [m]...Grain size after annealing,

d<sub>0</sub> [m]...The initial grain size,

A[m]...Pre-exponential factor,

t [s]...The duration of annealing,

Q [[.mol-1]...The activation energy,

T [K]...The annealing temperature [17].

R is the universal gas constant with the value of 8.314 J.mol<sup>-1</sup>K<sup>-1</sup>. Using this model, the value of activation energy of 131 kJ.mol<sup>-1</sup> was calculated.

Based on this empirical equation, the microstructure of the exposed valve in selected parts (Figure 4) was compared in terms of the grain size with the model samples. Equation (1) allowed to estimate the temperature on the exposed sample. The results are collected in Figure 5 and Table 3. The results are in relatively good agreement with the widely accepted mathematical model [17]. The temperature estimated by the model for our "B" area is almost identical. However, there is a discrepancy regarding "A" position. Ref. [17] predicts lower temperature than in "A" area, but our experimental results indicate a noticeably higher one. When considering the conditions in the engine, it can be seen that area "B" is flushed by combustion gasses during exhaust period of the engine, while the "A" area is fully exposed in the combustion chamber during the whole period of operation of the engine. On the other hand, there is the contact of the valve face and cylinder head seat insert, which is water-cooled. It indicates that the water cooling is not

sufficient to lower the temperature of the valve face in the case of this engine and therefore, the published model does not describe the behavior of this engine well. There could be some inaccuracy in the input data (estimation that 70 000 km corresponds to 1000 h of operation of the engine) and also slight local differences in the grain size caused by forming process, causing that the values of the temperature are affected, but the trend is clearly visible.

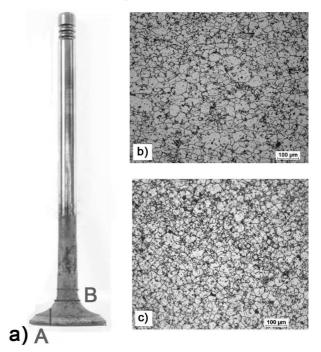


Fig. 5 Used exhaust valve scheme (a), microstructure in the area "A" (b), microstructure in the area "B" (c), electrolytically etched by oxalic acid

**Tab. 3** Grain size of used exhaust valve in areas shown in Figure 5 and corresponding temperatures calculated by equation (1)

State	Grain size (µm)	Calculated temperature (°C)
A	$53 \pm 8$	912
В	46 ± 6	752

## 4 Conclusion

This work showed that it is possible to determine the temperature on the exhaust valve made of austenitic heat-resistant steel using the microstructure evaluation of the exposed valve and its comparison with model samples of the same material annealed on known temperatures. We found that the values of temperature are correctly determined for the fillet in the area, where it is in contact with the combustion exhaust gasses. The temperature on the disc is higher than predicted by the models published in the literature. For the intake valves made of common martensitic valve steel Silchrome 1, this metallographic procedure according to ASTM E112-13 method fails due to the inability to visualize the grain boundaries. The results of this work can be used either in optimization of the construction of engines, or in the design of the valves both for primary production and aftermarket.

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#### References

- [1] ZHOU, H., JING, X., HE, S., LI, L. (2016) Research on Integrated Optimization Design Method for Diesel Engine Valve Train. In: Manufacturing Technology, Vol. 16, pp. 1398-1404.
- [2] ATES, S., ASLAN, O. ., TÜMER, M., ARISOY, C.F. (2024) Impact sliding wear behavior of Stellite 6 and Stellite 12 hardfacings. In: *Materials Chemistry and Physics*, Vol. 313, 128762.
- [3] MRÁZ, J., BAKALOVA, T. (2024) The Impact of Cryogenic Temperatures on the Hardness and Tribological Properties of Cobalt Alloys. In: *Manufacturing Technology*, Vol. 24, pp. 626-35.
- [4] LIU, L., ZHANG, L., YANG, D., ZHANG, Q., YU, D., CHEN, L. (2020) Two-phase flow-based numerical investigation of the temperature maps of sodium-cooled exhaust valves in a turbocharged engine. In: *Applied Thermal Engineering*, Vol. 181, 115977.
- [5] LOU, M., ALPAS, A.T. (2019) High temperature wear mechanisms in thermally oxidized titanium alloys for engine valve applications. In: *Wear*, Vol. 426-427, pp. 443-453.

- [6] SIVAKUMAR, E.R., KRISHNA, R., SENTHILKUMAR, P., KRISHNAKUMAR, T.S., COLAK, I. (2023) Comprehensive study of diesel engine characteristics of valves coated by titanium nitride (TiN). In: Proceedings of the Institution of Mechanical Engineers, Part E: Journal of Process Mechanical Engineering. doi:10.1177/09544089231157950.
- [7] NOVÁK, P, NOVÁ, K, VANKA, T, PRŮŠA, F. (2018) High-temperature Behaviour of New Fe-Al-Si Alloy Produced by Powder Metallurgy. In: *Manufacturing Technology*, Vol. 18, No. 2, pp. 299-302.
- [8] LARSON, J., JENKINS, L., NARASIMHAN, S., BELMORE, J. (1987) Engine Valves—Design and Material Evolution. In: *Journal of Engi*neering for Gas Turbines and Power, Vol. 109, pp. 355–361.
- [9] PIERCE, D., HAYNES, A., HUGHES, J., GRAVES, R., MAZIASZ, P., MURALIDHARAN, G., SHYAM, A., WANG, B., ENGLAND, R., DANIEL, C. (2019) High temperature materials for heavy duty diesel engines: Historical and future trends. In: *Progress in Material Science*, Vol. 103, pp. 109–179.
- [10] Lopes Silva, D.A., de Oliveira, J.A., Padovezi Filleti, R.A., Gomes de Oliveira, J.F., Jannone da Silva, E., Ometto, A.R. (2018) Life Cycle Assessment in automotive sector: A case study for engine valves towards cleaner production. In: *Journal of Cleaner Production*, Vol. 184, pp. 286-300.
- [11] MOGFORD, R., BALL, F. (1955) Exhaust valve life: Some metallurgical and mechanical considerations, In: *Proceedings of the Institution of Mechanical Engineers: Automobile Division.* Vol. 9, No. 1, pp. 147-169.
- [12] YU, Z.W., XU, X.L. (2006) Failure analysis and metallurgical investigation of diesel engine exhaust valves, In: *Engineering Failure Analysis*, Vol. 13, pp. 673-682.
- [13] YONG, M.F., KU, P.X. (2023) Study on the performance characteristics and failure analysis of exhaust valves. In: *Journal of Physics: Conference Series*, Vol. 2523, 012047.

- [14] HÖNIG, V, ORSÁK, M. (2016) Wear of Engine Oils Using Gaseous Fuels. In: *Manufacturing Technology*, Vol. 16, No. 5, pp. 923-927.
- [15] HÖNIG, V. (2018) Specifications of Wear of Oils in Two-Fuel Systems. In: *Manufacturing Technology*, Vol. 18, pp. 567-71.
- [16] HÖNIG, V, MIHOLOVÁ, D, ORSÁK, M. (2014) Measurement of Wear Metals in Engine Oils by Atomic Absorption Spektrometry Method. In: Manufacturing Technology, Vol. 14, No. 3, pp. 317-322.
- [17] CERDOUN, M., KHALFALLAH, S., BENIAICHE, A., CARCASCI, C. (2020) Investigations on the heat transfer within intake

- and exhaust valves at various engine speeds, In: *International Journal of Heat and Mass Transfer*, Vol. 147, 119005.
- [18] Standard Test Methods for Determining Average Grain Size, ASTM E112-13 (2021) https://img.antpedia.com/stand-ard/files/pdfs\_ora/20211231/ASTM%20E11 2-13(2021).pdf
- [19] XU, D., JI, C., ZHAO, H., JU, D., ZHU, M. (2017) A new study on the growth behavior of austenite grains during heating processes, In: *Scientific Reports*, Vol. 7, 3968.