IMAGE ANALYSIS OF TITANIUM CARBONITRIDES

[1]Petr MARTÍNEK, [2]Pavel PODANÝ

[1]*COMTES FHT a.s., Dobřany, Czech Republic, EU, petr.martinek@comtesfht.cz*

[2]*COMTES FHT a.s., Dobřany, Czech Republic, EU, pavel.podany@comtesfht.cz*

https://doi.org/10.37904/metal.2020.3515

Abstract

A manufacturer of crankshafts uses steel stock purchased from several vendors. The steels differ in their behaviour during the manufacture of the crankshaft, although they are the same grade. Some steel stock batches were repeatedly associated with much faster wear of grinding wheels than others. What is more, the crankshafts also suffered damage in production. As a result, the cost of production increased to unacceptable levels.

Titanium precipitates were suspected to be the cause of the grinding wheels going dull. Therefore, an investigation was carried out to determine in which phases the titanium addition is present. The quantities, sizes, distributions and morphologies of particles containing titanium were measured using image analysis. Finally, the data were correlated to the method of steel production, the vendor and the titanium content.

Keywords: Image analysis, titanium particles, machinability

1. INTRODUCTION

The manufacturer of crankshafts purchases stock from several vendors. The steels from individual batches differ in their behaviour during finish grinding, despite being identified as the same steel grade. With some batches of steel stock, grinding wheels wore much faster than with others and required shorter intervals between redressing, which increased the production costs. When a dull grinding wheel is used for grinding a crankshaft, it causes damage to the surface. Crankshafts are made by closed-die forging and machining. Finally, local induction hardening and grinding is performed on those locations which will experience severe loads in service. After the grinding operation, some crankshaft series repeatedly exhibited surface damage. The surface became overheated which sometimes led to surface cracking.

As the steel grade and the production route were identical for all crankshafts, the causes of these issues were investigated. One hypothesis was that the surface damage was due to excessive wear of grinding wheels caused by titanium-containing precipitates. This steel grade is alloyed with titanium in order to prevent grain coarsening during forging. The purpose of this investigation was to identify the phases in which titanium is present in this steel, to characterize the morphology, sizes and distribution of their particles and compare these aspects among the vendors.

2. METHOD AND SCOPE OF EVALUATION OF TITANIUM-CONTAINING PARTICLES

Titanium particles were characterized in eighteen specimens from six different vendors. All the specimens were from the same steel grade (**Table 1**). Titanium particles in the matrix were either isolated or found close to manganese sulphides (**Figure 1**). They were nitrides or carbonitrides. They also contained some vanadium and/or niobium (**Figure 2**).

Generally, titanium combines with nitrogen and carbon to form highly-stable nitrides, carbides and carbonitrides [1,2]. Large cube-shaped TiN particles may precipitate as early as in the liquidus region. At elevated temperatures, TiN particles are the most stable ones. They may form during the last solidification stage or just afterwards. The mechanism of this formation is segregation in interdendritic regions of the melt [3,4].

Table 1 Specified chemical composition of the steel grade supplied [wt%]

Element	C	Si	Mn	P	S	Cr	V	Al	N	Ti
Min	0.32	0.15	1.20		0.045		0.08	0.010	0.010	0.015
Max	0.41	0.80	1.60	0.025	0.065	0.30	0.20	0.030	0.020	0.035

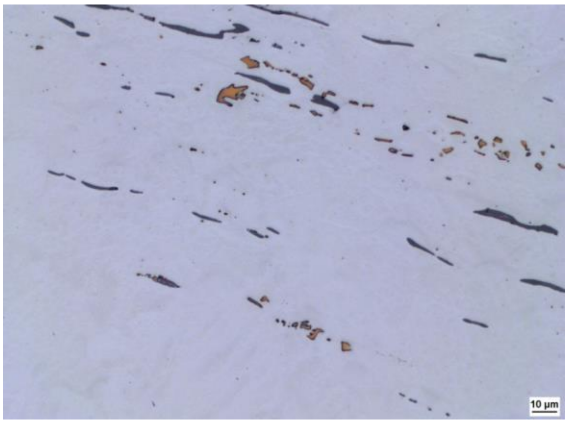

Figure 1 Precipitates containing titanium and manganese sulphide inclusions

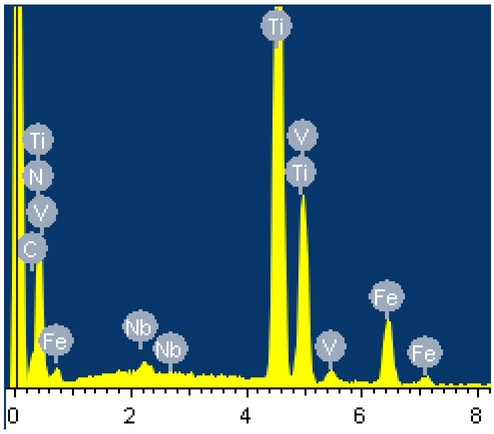

Figure 2 Energy spectrum of titanium carbonitride collected by EDX

Titanium-containing precipitates were studied using an optical microscope and the NIS Elements 3.2 image analysis software which is suitable for examination of titanium precipitates [5]. Longitudinal metallographic sections were prepared in identical locations of all crankshafts. Six regions were evaluated in each crankshaft. In each region, 100 micrographs were taken using Carl Zeiss – Observer.Z1m optical microscope. Analysis was then conducted on only 50 micrographs distributed in a chessboard-like manner. The area under evaluation was thus relatively large in order to reduce the influence of the non-uniform distribution of titanium particles in the material. The characteristics of interest included the area, circularity and number of titanium particles. For this evaluation the lower limit of a feature's area was set at 1 µm^2. This means that particles with an area smaller than 1 µm^2 were excluded from the evaluation. The rationale was to eliminate artefacts (small regions of several pixels identified by possibly inaccurate thresholding) and keep the quantitative evaluation accurate. Using a scanning electron microscope, large numbers of titanium carbonitrides were revealed whose size was under 1 µm but they were excluded from the analysis.

As mentioned above, the evaluation covered six regions in each crankshaft. In each of the regions, 50 micrographs at magnification 500 were examined. In total, 300 fields of view were captured from each crankshaft for evaluation, comprising an area of 10.9 mm^2.

3. RELATIONSHIP BETWEEN TITANIUM CONTENT AND QUANTITY OF PARTICLES

The prescribed range of titanium content for this material is 0.015 to 0.035 %. Some of the materials under examination had titanium levels of 0.020 ± 3 %, others had reduced titanium levels at 0.07 % and yet others had no titanium addition. Hence, the eighteen specimens were classified into three groups by their titanium content. In the first group, the titanium level was approximately 0.020 %. It comprised twelve specimens, becoming the largest group of the three. In the second group (four specimens), the titanium level was 0.007 %.

The third group had two specimens with titanium levels under 0.001 %, which is a negligible amount from the engineering viewpoint.

The numbers of titanium particles vary greatly, even between specimens with similar or identical titanium levels. Results for the third group are not adequately relevant, as the group only comprised two specimens. Despite these limitations, average quantities of particles for individual groups, regardless of the vendor, were calculated (**Table 2**).

Table 2 Calculated average quantities of titanium particles

Group #	Ti level [%]	Average quantity of all particles	Average quantity of particles sized > 10 μm^2	Average quantity of particles sized > 40 μm^2
1	0.02	475	48	3
2	0.007	147	18	0.25
3	< 0.1	7	2.5	0

The difference between the titanium levels of 0.020 % and 0.07 % was reflected in the average quantities of all titanium particles: 475 and 147, respectively. Three times lower titanium content translated into approximately three times fewer particles. In the specimens with minimal titanium levels, the quantity of particles was negligible.

When particles larger than 10 μm^2 were considered, the results varied more significantly. In group 1 with titanium levels of 0.020 %, the average number of particles of this size was 48. In group 2, the average number of particles was 12. For particles sized above 10 μm^2, the ratio of these numbers for groups 1 and 2 was four. The difference between quantities of particles of this size was greater than the difference in the titanium levels. In group 3, the quantity of particles was much smaller than in the previous two,

The average number of particles larger than 40 μm^2 in specimens of group 1 with 0.020 % titanium is 3. In group 2, only one such particle was found in one specimen, which makes 0.025. With particles larger than 40 μm^2, the number of particles changes much more steeply with the amount of titanium. In group 3, there are no particles of this size.

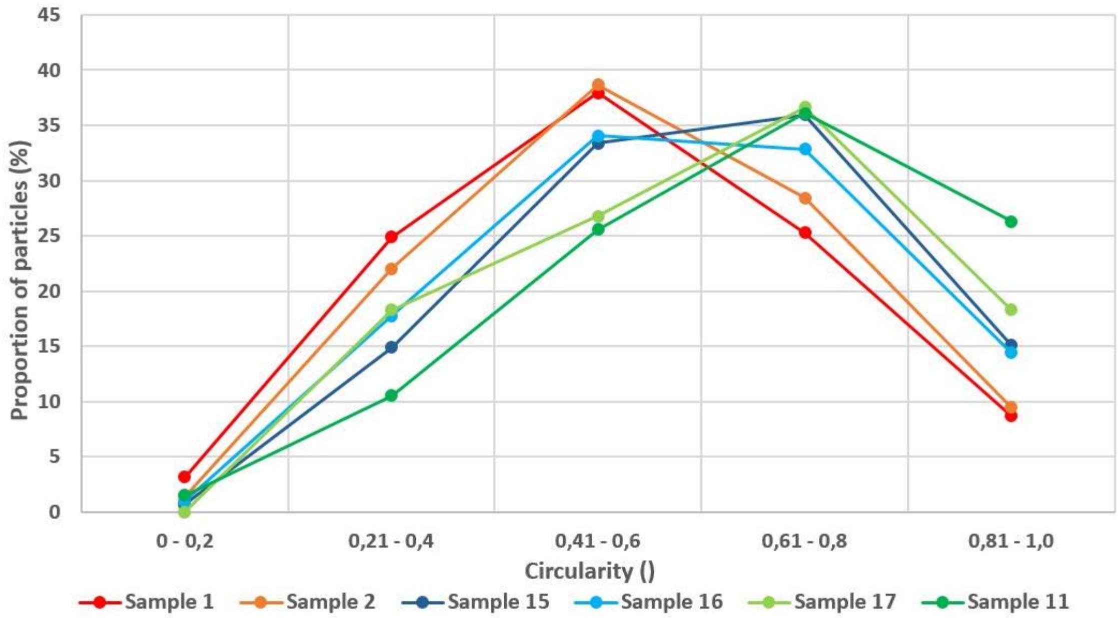

Figure 3 Proportions of particles within intervals of circularity

4. PARTICLE MORPHOLOGY

One of the particle characteristics evaluated in this investigation was morphology. For this purpose, six representative specimens were chosen from the entire set. The materials of specimens 1 and 2 (plotted in red in the graph) were produced by the basic oxygen process. The amount of titanium was about 0.020 %. Specimens 15 and 16 (data plotted in blue) were taken from electric furnace steels. Their titanium levels were approximately 0.020 %. Specimens 17 and 11 (green) were taken from electric furnace steels as well but their titanium level was 0.007% (**Figure 3**).

Basic oxygen steels contained the largest proportion of particles with low circularity, i.e. with less favourable morphology. By contrast, electric furnace steels showed better morphology. Lower amounts of titanium were favourable because specimens with 0.007 % Ti had the highest share of particles with higher circularity. This is probably due to the absence of coarse particles which generally possess lower circularity (**Figure 3**).

5. VARIANCE IN PARTICLE SIZE

One of the parameters under examination was the maximum area of titanium particles in individual specimens. The same six specimens as in the preceding section were used for this measurement.

The results are plotted in **Figure 4**. The graph shows the variance of maximum areas of particles for individual regions of measurement. The red points indicate the average maximum area in individual regions in different specimens. It is clear from the graph that the first four specimens, which contained approximately 0.020 % titanium, showed much larger variance than specimens 17 and 11 which only contained 0.007 % Ti. Even the averages of maximum values are lower in these specimens than in the previous ones. The main reason is the absence of very coarse particles in specimens with very low titanium levels. As stated above, the specimens with 0.007 % and less titanium are virtually free from particles sized above 40 μm^2. In the chosen representative specimens, this limit is even less: 30 μm^2.

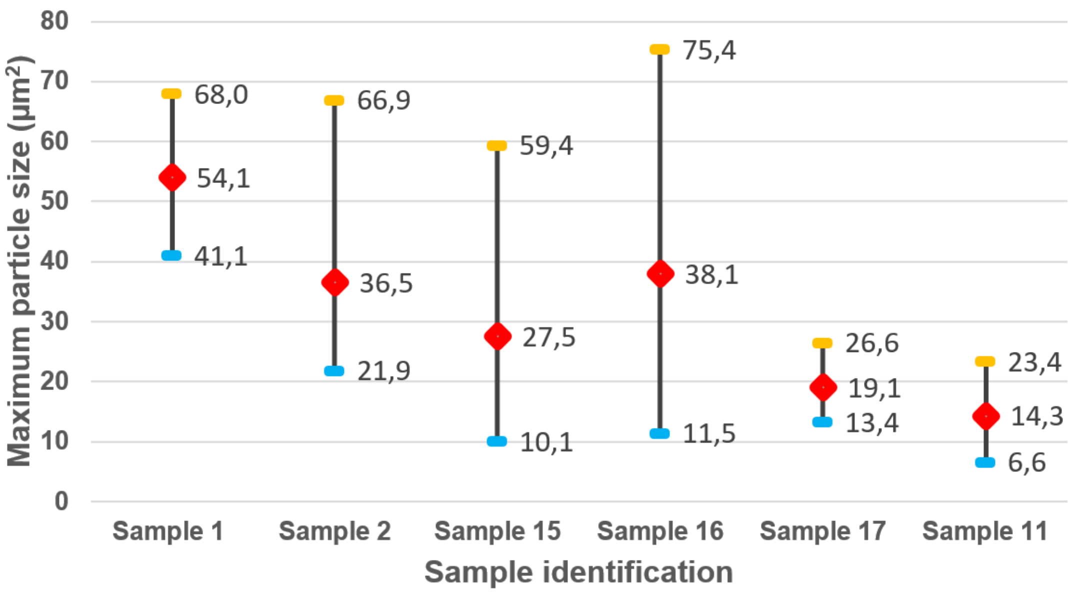

Figure 4 Variance of maximum sizes of particles in selected specimens

6. EFFECT OF STEELMAKING METHOD

Steels for crankshafts are produced using two processes. One is the basic oxygen process and the other takes place in electric furnaces. An analysis was conducted to identify the impact that the steelmaking process has on titanium particles.

All specimens from the above vendors which had the same titanium content, i.e. about 0.020 %, were considered. Average numbers of titanium particles were calculated for different sizes in steels from individual vendors and for different steelmaking methods. The findings are summarized in **Table 3**.

In electric furnace steels, there are more particles, regardless of their size, for both vendors. However, above particle size 10 μm^2 and 40 μm^2, respectively, there is a change in the trend and more particles are found in basic oxygen steels. The conclusion is that electric furnace steels have better-sized titanium particles. These steels contain large numbers of particles overall, where these particles can impede grain growth. Besides, they contain fewer coarse titanium particles which have less impact on grain size.

Table 3 Average numbers of titanium particles in different size intervals

Production method	Vendor	Number of all particles		Number of particles sized > 10 μm^2		Number of particles sized > 40 μm^2	
Basic oxygen steel	1	253.0	448.1	72.0	51.6	12.0	3.4
	2	476.0		48.7		2.1	
Electric furnace steel	3	552.0	528.3	54.0	40.0	2.0	1.8
	4	520.3		35.3		1.7	

7. EFFECTS OF METALLURGICAL PROCESS

The sizes, amount, and distribution of particles are also governed by the quality of the metallurgical process. Specimens with the same titanium content exhibit marked differences in all titanium particle characteristics, which include specimens from the same manufacturers. One example is the comparison between specimens 2 through 5 from vendor 2. All of them contain approximately 0.020 % titanium. Yet, with specimens 2 and 3, grinding wheels had to be redressed after grinding 30 to 50 crankshafts. On specimens 4 and 5, the number of crankshafts which were ground between redressing operations was 120. In specimens 4 and 5 (with good machinability), the number of particles was 14.3 % less than in specimens 2 and 3. This difference becomes even greater when particles with area of no less than 10 μm^2 are considered. Then the difference between OK and NOK specimens becomes 30 %. This is significant enough to induce different behaviour in crankshafts being ground.

It is reported that above 0.05 % titanium, sharp-edged TiC carbides form along with non-uniformly-distributed TiN nitrides. The latter float up in molten steel and begin to segregate [4]. To prevent this, steel should be poured rapidly and additions applied to the melt appropriately.

Table 3 Number of particles vs. machinability

Specimen	Sample 2		Sample 3		Sample 4		Sample 5	
Ti level	0.020		0.021		0.019		0.020	
Machinability	NOK: 30-50 workpieces between redressing				OK: 120 workpieces between redressing			
Indicator	Quantity of particles	Proportion of particles [%]	Quantity of particles	Proportion of particles [%]	Quantity of particles	Proportion of particles [%]	Quantity of particles	Proportion of particles [%]
Total	559	100.0	361	100.0	462	100.0	326	100.0
above 10 μm^2	77	13.8	59	16.3	56	12.1	39	12.0
above 40 μm^2	4	0.7	2	0.6	3	0.6	3	0.9

8. CONCLUSION

A manufacturer of crankshafts reported repeated burning of ground workpiece surfaces due to dull grinding wheels, which resulted in damage to the products. The suspected cause of the wheels going dull was titanium particles, as the steel is alloyed with titanium. The goal of this investigation was to evaluate titanium carbonitrides using image analysis and compare the results for different steel vendors.

The quantities of particles found in individual specimens indicate that a two-third difference in titanium content (0.020 % vs 0.007 %) leads to the same difference in the total number of all titanium particles (by approximately two thirds). The total number of titanium particles is directly proportional to the amount of titanium. With decreasing titanium content, the quantity of the coarsest particles decreases even faster. In fact, no particles larger than 40 μm^2 are present eventually.

This is corroborated by data on maximum sizes of particles. With titanium level of about 0.020 % the variance in the maximum size of particle is much larger than in specimens which contain a mere 0.007 % Ti. Even the averages of maximum values are lower in these specimens. The main reason is the absence of very coarse particles in specimens with very low titanium. Specimens with 0,007% titanium or less are practically free from particles larger than 40 μm^2. Those above 30 μm^2 are very rare.

This shows that the titanium level and the size of particles affect the particle morphology. Specimens with 0,007 % Ti contain larger quantities of particles with higher circularity than those with 0.020 % titanium. This is probably due to the absence of coarse particles which generally possess lower circularity. Coarse sharp-edged particles are believed to have greater impact on grinding wheel dulling than round particles.

An assessment of particle morphology showed that steels made by the basic oxygen process contained larger proportion of particles with low circularity, i.e. with less favourable morphology. By contrast, electric furnace steels showed better morphology of particles. This is due to the mean size of particles formed as a result of different production methods. It was demonstrated that in electric furnace steels, there are more particles, regardless of their size. However, above 10 μm^2 and 40 μm^2, respectively, there is a change in the trend and more particles are found in basic oxygen steels.

The parameters vary greatly among the specimens, even between those from the same manufacturer and with the same titanium levels. These variations are likely due to the quality of the metallurgical processing. The pouring speed and the quality of the alloy addition step may affect the utility properties of steel substantially.

ACKNOWLEDGEMENTS

The paper was supported from ERDF Research of advanced steels with unique properties, No. CZ02.1.01/0.0/0.0/16_019/0000836.

REFERENCES

[1] CEJP, J., MACEK, K. Vliv tepelného zpracování na houževnatost mikrolegovaných nízkouhlíkových ocelí. In *METAL 2002: 11th International Conference on Metallurgy and Materials.* Ostrava: Tanger s.r.o., 2002.

[2] PARDO, A., et al. Influence of Ti, C and N concentration on the intergranular corrosion behaviour of AISI 316Ti and 321 stainless steels. *Acta Materialia*. 2007, vol. 55, pp. 2239–2251.

[3] ZHOU, C., PRIESTNER, R. The Evolution of Precipitates in Nb-Ti Micro alloyed Steels during Solidification and Post-solidification Cooling. *ISIJ International*. 1996, vol. 36, pp. 1397-1405.

[4] PANDIT, A. Strain induced precipitation of complex carbonitrides in Nb V and Ti V icroalloyed steels. *Scripta Materialia.* 2005, vol. 53, no. 11, pp. 1309–1314.

MARTÍNEK, P., NACHÁZEL, J., PODANÝ, P. Decreasing of carbonitrides size and content in austenitic steel by means of heat treatment and thermomechanical processing. In: *Materiali in tehnologije* (Materials and Technology), 49. [online]. LJUBLJANA: IMT Ljubljana, 2015. pp. 31-36. ISSN 1580-2949