# MACROSCOPIC METHOD FOR ASSESSING THE CONTENT OF NON-METALLIC INCLUSIONS - BLUE FRACTURE

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#### SUMMARY

Non-metallic inclusions usually deteriorate mechanical properties and surface quality of steel products. Presence of non-metallic inclusions might have negative affected some of the mechanical properties of steels like ductility, toughness, anisotropy, and formability. Macroscopic method for assessing the content of non-metallic inclusions in forged or rolled steel products is used for determination non-metallic inclusions visible to the naked eye or with aid of a magnifying with magnification of not more than 10 times. Only inclusions equal to or greater than 1 mm long are taken into consideration. In this paper is given procedure for blue fracture test method applied on bar diameter  $\phi$ 60 mm bearing steel 100Cr6.

# 1. INTRODUCTION

Low alloy steel, 100Cr6 thanks to its high hardness, wear resistance, surface finishing and dimensional precision, it is widely used to manufacture mechanical components, such as precision bearings, automotive components (brakes, steering, line shaft), bicycle, agitators, appliances, sliders, quick couplings, machine tool, lock mechanisms, conveyor belts, skates, pens, pumps, castors, measurement instruments, valves. The presence of non-metal inclusions in the steel 100Cr6 should be minimized due to the exploitation conditions of the specified components.

# 2. ORIGIN AND PROPERTIES OF NON-METALLIC INCLUSIONS

During produce any type of steel, it is impossible to avoid the appearance of non-metallic inclusions. Non-metallic inclusions can not be separated during the crystallization process and in solid alloy act as a harmful side of the body.

Non-metallic inclusions in steels can be divided into two groups, those of indigenous and those of exogenous origin. The former group contains inclusions occurring as a result of the reactions taking place in the molten or solidifying steel, whereas the latter contains the inclusions resulting from mechanical incorporation of slags, refractories or other materials with which the molten steel comes into contact. Exogenous inclusions are usually larger than the indigenous inclusions and, thus, non-metallic inclusions can also be divided into microinclusions and macroinclusions. Macroinclusions are more detrimental when their effects on the properties of steel, and especially fatigue properties, are considered [1].

Non-metallic inclusions are chemical compounds of metals (e.g. iron, manganese, aluminium, silicon and calcium) with non metals (e.g. oxygen, sulphur, carbon, hydrogen and

nitrogen). Non-metallic inclusions form separate phases. The non-metallic phases containing more than one compound (e.g. different oxides, oxide + sulphide) are called complex non-metallic inclusions (spinels, silicates, oxy-sulphides, and carbonitrides).

Despite the presence of non-metallic inclusions in steels in small percentage (0.01% to 0.02%), they have a significant effect on the properties of steels [2]. They are the cause for dangerous and serious material defects such as brittleness and a wide variety of crack formations. However, some of these inclusions can also have a beneficial effect on steels properties by nucleating acicular ferrite during the austenite to ferrite phase transformation especially in low carbon steels. The properties of steels generally affected by the non metallic inclusions include tensile strength, deformability or ductility, toughness, fatigue strength, corrosion resistance, weldability, polishability, and machinability [3].

Some inclusions found in bearing steels have incoherent interfaces with the steel and the stress described is that tangential to the inclusion-matrix interface. All of the inclusions listed melt at a temperature greater than that of the steel, and are less dense compared with the steel. Some of inclusions which are listed in Table 1, are problematic in bearing steels for a variety of reasons. They have different thermal expansion coefficients from the matrix, in most cases less than that of austenite. As a consequence of cooling to ambient temperatures, tensile and compressive residual stresses develop parallel and normal to the inclusion-matrix interface. The inclusions may be brittle, and the resulting cracks concentrate stress and hence may propagate into the steel. Even the simple presence of an uncracked inclusion introduces a mechanical heterogeneity which locally changes the distribution of stress. Almost all of the common non-metallic inclusions have weak interfaces with the steel. Strain incompatibilities during deformation processing of the steel can then lead to the formation of cavities at the localized at the inclusion interface along the direction of the principle plastic strain [4].

Inclusion	Shape	Hardness (HV30)	$e_{\rm T}  (10^{-6}  {\rm K}^{-1})$
MnS, CaS	Streaky	150-170	18.1
$Al_2O_3$	Aggregates	pprox 2200	8.0
Ca aluminates	Globular	900-2500	6.5 - 10.0
MnO-SiO <sub>2</sub>	Oblong	> 1100	5.0
TiC	Sharp cuboids	$\approx 3000$	9.4
TiN	Sharp cuboids	$\approx 2500$	9.4

Table 1. Properties of non metallic inclusions [4]

Debonding can occur during the course of fatigue. Alumina inclusions have been shown to detach from the matrix during loading, thus concentrating stress in the surrounding matrix; in contrast, titanium carbonnitride particles are apparently strongly connected to the matrix and hence fracture by cleavage, leading to a different mechanism of crack initiation [4], Fig. 1.



Figure 1. (a) Detachment of matrix from spherical alumina inclusion in a push–pull fatigue test. The cusps on the cavity are assumed to be associated with the plane of hot-rolling. (b) Similar effect but the cavity illustrated is empty (c) Cleaved Ti(C,N) particle which maintains a bond with the matrix. Micrographs courtesy of H. Bomas [4].

### 2.1. Influence of non-metallic inclusions

The presence of non-metallic inclusions in a sense represents the crack or cavity in the steel as a metal connection interruption basis weight and acts as the internal notch. Considering the strength of the inclusions is small and that the bond between the inclusions and the metal barely exists [3], this means that non-metallic inclusions can only transmit insignificant strains.

Non-metallic inclusions are no coherent with metallic matrix, they prevent dislocation migration and serve as stress concentrators. Due to this reason, 70...100% primary microcracks were initiated on non-metallic inclusions, inspite of only 0.1- 0.2 volume percent of inclusions existing in steel. It was established that microcracks contours were often faithful copies of inclusion contours, contributing greatly to crack formation in steel in process of cyclic loading [5].

Since the latter are products of reactions within the steel, they are normal constituents of it. Because ordinary manufacturing processes cannot entirely rid the steel of such inclusions, it is desirable to control their kind and amount within such limits that the steel is relatively free from those inclusions which are considered most injurious [6].

#### 2.2. Techniques of Evaluating Macro Inclusions

Unlike micro inclusions that are dominant in steel, macro inclusions are very rare and their detection requires more time and attention if using the conventional metallographic methods. Very skilled SEM and LOM users waste more time on samples and in most cases these inclusions are not even detected. With advancement in the ladle metallurgical processes, very low oxygen contents can be achieved in steel and this has resulted to high cleanliness in steel products. The index for macro inclusions has therefore been reduced to zero in recent years by using both step down tests and blue fracture testing. It has becoming more and more difficult to detect macro inclusions even though they exist in steel products. Reclamations are received by clean steel companies each year and macro inclusions have been the contributing factor for some failures [6].

There are few methods for determination macro non-metallic inclusions [7, 8]:

- Ultrasonic immersion test,
- Magnetic particles
- Blue fracture test,
- Step-mashined test,
- Hardness fracture test.

#### 2.3. Makro inclusions

Operations such as deoxidation and desulfurization are done in order to obtain the correct amounts of oxygen and sulphur in the steel. These elements contribute to the formation of non-metallic inclusions in steel during solidification and the consequences of these inclusions are catastrophic to the mechanical properties of the material during usage. Elements such as Al, Si, Ca and the Rare Earth Metals (REM) are used to remove oxygen and sulphur from steel. Micro inclusions (1-20  $\mu$ m) that may formed from the reactions of these elements often coagulate to form clusters and inclusions of bigger sizes (> 100  $\mu$ m) known as macro inclusions [9].

# 3. BLUE FRACTURE TESTING

Blue fracture testing of macro non-metallic inclusions may be carried out quantitatively or qualitatively. Quantitative examination is carried out by counting the inclusions and using one or both of the following parameters of the inclusions: length and thickness.

The distribution of the inclusions based on length is given in table 2.

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Symbol	Length <i>l</i> (mm)
LO	No macroscopic inclusion
L1	$1.0 \le l \le 2.5$
L2	$2.5 \le l \le 5$
L3	$5.0 \le l \le 10$
L4	1 > 10

Table 2. Inclusion distribution based on length [7]

The distribution of the inclusions based on thickness is given in table 3.

Symbol	Thickness <i>e</i> (mm)
T0	No macroscopic inclusion
T1	$0.1 \le e \le 0.25$
T2	$0.25 \le e \le 0.50$
T3	$0.5 \le e \le 1.0$
T4	e > 1.0

 Table 3. Inclusion distribution based on thickness [7]
 Inclusion distribution based on thickness [7]

Qualitative examination is carried out by comparison with the series of ten reference diagrams, Figure 2. Account is taken of the positions of the inclusions within the section for example core, surface or uniform distribution,



Explanation of terms: Very short stringers: 1 to 2.5 mm Short stringers: > 2.5 mm Long stringers: > 5 mm

Few:  $\leq 3$ Several: > 3Thick: > 0.5 mm

Figure 2. Standard diagrams for the blue fracture test method [7]

# 4. MATERIAL FOR TESTING

For this paper work, wrought bar diameter  $\phi$  60 mm bearing steel 100Cr6 was tested. Chemical composition is given in Table 4.

Material	Chemical composition, %					
	С	Si	Mn	Р	S	Cr
100Cr6	0.85	0.56	1.10	0.011	0.014	1.47

Table 4. Chemical composition of bar  $\phi$  60 mm

# 5. EXPERIMENTAL PART

Blue fracture testing was done at Metallographic and Mechanical laboratories of Institute "Kemal Kapetanović" of Zenica.

#### 5.1. Sampling of test piece

Test pieces are slices with thickness of 10 mm. The thickness being measured parallel to the longitudinal direction. For this paper work three samples were tested. The test pieces for blue fracture testing are given at Figure 3.



Figure 3. Test pieces for blue fracture testing

# 5.2. Preparation of test piece

Both sides of the test piece is prepared to get plan-parallel surfaces. At the center of the normal side of the longitudinal axis of the rod, a groove was formed. The purpose of this groove is to facilitate the fracture of the test piece. The groove depth is up to 2 mm. Test pieces were heated in laboratory furnace so that at moment of starting the test, the metal is at blue brittleness temperature (350-400 °C). After heating samples fractured with the testing mashine AMSLER, 200 kN in the Mechanical laboratory, (Fig. 4a). Broken samples is shown at Fig. 4b.



Figure 4. AMSLER - Testing mashine and broken sample

# 5.3. Testing procedure

One of two broken parts of test piece is examined with magnification less then to X 10 using stereo light microscope TECHNIVAL 2. Testing results are given in Table 5. The broken surface is compared to diagram chart given at Figure 2.

Sample of 100Cr6	Thickness of non- metallic inclusion (mm)	Length of non- metallic inclusion (mm)	Testing area (dm <sup>2</sup> )	Total length of non-metallic inclusion per testing area
1	0.20	1.5	0.035	
2	0.20	1.0	0.035	$4.3 \text{ mm} / 0.112 \text{ dm}^2$
3	0.25	1.8	0.420	

*Table 5. Blue fracture testing of bar*  $\phi$  *60 mm* 

In accordance to diagram chart given at Figure 2 given in standard BAS ISO 3763:2009 the assessment is: 1.

Macro non-metallic inclusion observed on one of the test piece is given at Figure 5.



Figure 5. Macro non-metallic inclusion in blue fracture

#### 6. CONCLUSION

Based on the performed treatment heating at temperature of blue fracture and samples testing at X10 magnification, macro non-metallic inclusion, very short stringer, was detected. By the length and thickness of the macro non-metallic inclusion were estimated at 1.

Blue fracture as a method of testing macro non-metallic inclusions is relatively simple and reliable. The size of the detected non-metallic inclusions indicates the risk of application of the test batch and indicates the need for further testing due to possible influence of the presence of non-metallic inclusions on the mechanical properties of steel 100Cr6.

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