Effect of Diamond-Like Carbon Thin Film on the Fatigue Strength of AISI 4340 Steel

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Abstract: AISI 4340 is known as super strength steel and widely applied in military equipment, aircraft components, automotive components, drilling device and so on because of its excellent behavior in wear, corrosion, fatigue, high temperature, and high-speed operating conditions. However, due to continuing work which reduced the performance of that component surface during their service life, an effort to improve the surface properties for a longer service life should be carried out. This paper presents the research result of the influence of diamond-like carbon coating deposited using home-made *DC Chemical Vapor Deposition (DC-CVD)* on the surface of AISI 4340 steel. As a carbon source, a mixture of argon (Ar) and methane (CH₄) with a ratio of 24%: 76% was used in this experiment. The conditions of the experiment were 400°C of temperature at various gas pressures (1.2 mbar, 1.4 mbar, 1.6 mbar, 1.8 mbar, and 2.0 mbar) for 5 hours of coating time. Investigated surface properties are hardness, fatigue strength, and surface morphology. It was found that the optimum conditions in enhancing fatigue strength at 1.4 mbar of pressure. At these conditions, the fatigue strength increase from 401 MPa to 514 MPa, the microhardness increase from 327 VHN to 625 VHN. Based on surface morphology observation of the fracture surfaces, it shows that for raw material, an initiation crack starting from the surface. However, after being coated for 1.2 mbar, 1.4 mbar, and 1.6 mbar, the initial crack begins from the inside. The high hardness layer hinders the fatigue crack initiation.

Keywords: AISI 4340 Steel, Diamond-Like Carbon, Fatigue Strength, CVD

1. Introduction

AISI 4340 is N-Cr-Mo steel having high hardenability, exceptional tensile strength, high ductility and high toughness [1-2]. Because of its super properties, AISI 4340 is widely used for fabrication of parts components in automotive, aerospace as well as metal mechanics industries. In this sense, a piece as shafts, bolts, rods, reels, rotor, and gear can be fabricated from this alloy, because of its excellent mechanical properties. The lifetime of this alloy is not long enough because of its low corrosion and tribological resistance and poor tribological properties [3-4]. Surface treatments such as shot peening, deep rolling, nitriding, and carburizing or diamond-like carbon coating are known to be very effective for the protection of a surface against fatigue and crack

initiation. This protection due to surface hardening and residual compressive stresses introduced below the surface, therefore, the fatigue properties are improved by the surface layer [5-7]. Because of that characteristic, the surface treatment technique is widely used to improve fatigue, wear, and corrosion resistance of various industrial products.

The word fatigue comes from Latin word *fatigare* which mean “to tire” [8]. Fatigue is the most common type of failure in engineering structures. It estimated that 50 % - 90 % of mechanical failures are due to fatigue [8-12]. Most of the failures are unexpected and are caused by repetitive load cycles. To improve the fatigue strength of the material, one of the most used methods is a surface treatment of the materials in which hardness, wear resistance, corrosion resistance is improved [8].

Metal fatigue involves the initiation and growth of cracks

under the action of cyclic stresses caused by repeated application of service loads. Residual stresses remaining from fabrication also play an important role in aggravating metal fatigue. The phenomenon of fatigue is generally divided into three steps; (1) crack initiation, (2) crack growth/propagation, and (3) final fracture. Four parameters that influence the fatigue strength are; (1) stress difference, or as most often called stress range, (2) structural detail geometry, (3) material characteristic and (4) environment [13].

Fatigue is the condition whereby a material cracks or fails because of repeated (cyclic) stresses applied below the ultimate strength of the material. Fatigue failure often occurs quite suddenly with a catastrophic result. Fatigue strength is defined as the stress level, that a material can withstand to reach a defined number of cycles, generally enough to be considered as “infinite life”. In this case, the plot must specify the number of cycles at which the fatigue strength is reported. The maximum stress that material can endure without failure is called fatigue (endurance) limit. Below this stress level, there is a 50 % probability that no failure will occur on a material at the same condition. Fatigue properties of materials are often described using the fatigue limit or the S-N curve (fatigue curve, Wöhler curve). The S-N curve describes the relationship between cyclic stress amplitude and a number of cycles to failure [14].

Diamond-like carbon (DLC) films originated from hydrocarbon sources are carbon-based films that present a structure similar to diamond without the bond angles that exist in the tetrahedral diamond structure. Therefore, they are mainly amorphous and a certain amount of hydrogen [15]. DLC coating becomes special attention due to their properties such as low friction coefficient, high chemical stability, high hardness, optical transparency, high electrical resistivity, and low electron affinity. Thereby they can be used in optoelectronic devices, protective films for tribological or chemical applications, automotive parts, and tools, coating for dies or molds and biological parts. DLC films can be produced using Chemical Vapor Deposition (CVD) or Physical Vapor Deposition (PVD) technique. In this research we choose PVD technique, because we have been successfully designed and constructed a home-made D-C CVD with specification

diameter $D = 32$ cm, height of tube $h=40$ cm, and power supply 2 kW which usually used for plasma nitriding, plasma carburizing and plasma carbonitriding of metal or automotive components successfully [17-19].

The gas usually used in the formation of DLC film using glow discharge plasma is a mixture of a hydrocarbon gas with argon (Ar) or helium (He). Some of the most widely used hydrocarbon gases include methane (CH_4), ethane (C_2H_6), butane (CH_4H_{10}), and benzene (C_6H_6). In this case, CH_4 is more effective than C_2H_2 to form a denser film, and the results recommend that source gases with higher/C ratios exhibit superior friction and wear performance [16]. In this research, it is reported the effect of diamond-like carbon coating on the fatigue strength of AISI 4340 steel. For the purpose, as a carbon source, we use a mixture of argon (Ar) and methane (CH_4) with a ratio of 24%:76%. The conditions of the experiment were 400°C of temperature at various pressures (1.2 mbar, 1.4 mbar, 1.6 mbar, 1.8 mbar, and 2 mbar) for 5 hours of coating time.

2. Methodology

The sample material is the AISI 4340 steel with chemical compositions are shown in Table 1.

Table 1. Chemical Composition of the AISI 4340.

Element	Quantity (%)
C	0.38
Si	0.26
Mn	0.69
P	0.02
S	0.004
Cr	0.80
Mo	0.22
Ni	0.69
Fe	Bal.

Fatigue test was conducted using a rotating bending machine. The specimens were machined into the shape and size according to JIS Z 2274 standard as shown in Figure 1. While the schematic of home-made DC Chemical Vapor Deposition (*DC-CVD*) for deposition of diamond-like carbon is shown in Figure 2.

Figure 1. Fatigue test specimen according to JIS Z 2274 standard.

Figure 2. Schematic of home-made DC Chemical vapor deposition (DC-CVD) for deposition of diamond-like carbon.

3. Results and Discussion

Before fatigue test, the first step is to determine the tensile strength of the raw materials, and the test result is shown in Table 2. This data was used as a consideration in the determination of the nominal load in the fatigue test.

Table 2. Tensile Strength of Raw Materials of the AISI 4340 steel.

Element	1	2	3
Pyield	8840	8800	9000
Pmaks	9360	9340	9440
A0	80.4	79.3	81.2
L0	50	50	50
pyield	1077.5	1087.5	1086.2
pmaks	1141	1154	1139.3
Δl	7.73	7.46	6.56
ϵ	15.46	14.92	13.12

3.1. Hardness Test

The influence of pressure variation at 400°C of temperature and 5 hours coating time on the surface hardness of coated AISI 4340 steel is shown in Figure 3. It showed that for the higher pressure of the coating process the higher hardness and reached the optimum hardness around 625 VHN. This optimum hardness was achieved at 1.4 mbar of pressure. However, after 1.4 mbar, the hardness tends to decrease. Correlation between pressure and formed plasma during the coating process is the more pressure, the more formed ions/molecules. Formed ions during the coating process are C^+ , Ar^+ , and $CxHy^+$; these ions contribute to the formation of diamond-like carbon coating [9].

The rate of DLC coating formation is greatly influenced by the amount of forming ions and based on this research, it was found that the optimum condition (the highest hardness) was at 1.4 mbar of pressure (see Figure 3). After 1.4 mbar, the hardness tends to decrease. As the flow rate of the gas increases, the pressure of the plasma inside the chamber increases, this in turn increases the rate of generation of atomic hydrogen and methyl radicals; as a result, the growth rate of the film increases. However, increasing of Ar concentration above a certain level, the CH_4 concentration becomes so low thus the etching rate due to Ar becomes more pronounced than the deposition rate from CH_4 . As a result, the growth rate drops.

Figure 3. Effect of pressure at 5 hours of duration time on the surface hardness (HV) of AISI 4340 coated at 400°C of temperature. In uncoated condition (raw material) the VHN was 327 kg/mm².

Bombardment of Ar and H ions on the surface of the DLC film produces more dangling bonds due to sputtering of hydrocarbons and abstraction of H from the film, thus the incoming ad-atoms bonds with nucleated regions giving rise to larger grain size. Hall Petch stated that the strength of metallics equal to the frictional stress plus a factor (k) times the inverse of the square root of the grain size (d). Raising in the grain size will cause the material to become softer. Reducing the grain size will cause the material to become stronger. Grain size reduction is also mean to increase the

toughness of metal.

3.2. Fatigue Test

The fatigue test to failure was carried out in the air up to $N = 10^7$ cycles. Figure 4 shows the S–N curves of the uncoated and coated samples treated for various of pressure at 400°C of temperature and for 5 hours of deposition time. Table 3 shows the effect of pressure on the fatigue strength of AISI 4340 coated at 400°C of temperature and 5 hours of duration time.

Figure 4. S–N curves of the uncoated and coated samples treated for various of pressure at 400 °C of temperature for 5 hours of deposition time.

Table 3. The effect of pressure at 5 hours of deposition time on the fatigue strength of AISI 4340 coated at 400 °C of temperature.

Pressure	Fatigue strength
Raw	401 MPa
1.2 mbar	457 MPa
1.4 mbar	514 MPa
1.6 mbar	462 MPa
1.8 mbar	401 MPa
1.8 mbar	399 MPa

Figure 4 and Table 3 show the results of fatigue test for as-received specimens (raw materials) and coated specimens. It shows that the fatigue strength of as-received specimens is 401 MPa. After being nitrided for various of pressure, showed that for the higher pressure of the coating process, the higher fatigue strength and reached the optimum fatigue strength around 514 MPa, and this condition was achieved at 1.4 mbar of pressure. However, after 1.4 mbar of pressure, the fatigue strength tends to decrease. Increased fatigue strength after the nitriding process is caused by the increase of surface hardness

and the formation of compressive residual stresses in the surface during the coating process. The high hardness layer hinders the fatigue crack initiation. The compressive residual stress is superimposed to the external load and leads to a reduction of the effective stress in tension. Since only tensile stress produces fatigue cracks and contributes to crack propagation, a reduction of the effective stress in tension increases the fatigue strength.

3.3. Fatigue Fracture Surface Analysis

The fracture surface was observed using Scanning Electron Microscopy (SEM). Figure 5 shows the typical SEM images surfaces for as-received and DLC coated specimens. Based on SEM observation of the fracture surfaces shown that initiation crack starts from the surface. However, after being coated for 1.2 mbar, 1.4 mbar, and 1.6 mbar, the initial crack start from the inside. The high hardness layer hinders the fatigue crack initiation. The compressive residual stress is superimposed to the external load and leads to a reduction of the effective stress in tension. Since only tensile stress produces fatigue cracks and contributes to crack propagation, a reduction of the effective stress in tension increases the fatigue strength.

Figure 5. Crack initiation of as-received, coated at 1.2 mbar, 1.4 mbar and 1.6 mbar of pressure for 5 hours of time, the red arrow indicates crack initiation while the white arrow indicates beach mark direction.

4. Conclusion

The hardness of the as-received materials is 327 VHN, after being coated for various pressure and time; generally, the hardness increased and reached optimum in order of 625 VHN. This condition was achieved at 1.4 mbar of pressure and 5 hours of deposition time. In the rotary bending test, it was found that, for as-received materials, the fatigue strength is 400 MPa. After being coated for various pressure and time, the fatigue strength increased and reached optimum in order of 514 MPa. This condition was achieved at 1.4 mbar of pressure and 5 hours of deposition time. A surface morphology observation of the fracture surfaces shown that for raw material, initiation crack starts from the surface. However, after being coated for 1.2 mbar, 1.4 mbar, and 1.6 mbar, the initial crack starts from the inside. The high hardness layer hinders the fatigue crack initiation.

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