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**Coal-Fueled Diesel Technology Development
Nozzle Development for Coal-Fueled Diesel Engines**

Topical Report

**R. N. Johnson
M. Lee
R. A. White**

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**For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
P.O. Box 880
Morgantown, West Virginia 26507-0880**

**By
General Electric Corporate Research and Development
Schenectady, New York 12301**

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ABSTRACT

Direct injection of a micronized coal water mixture fuel into the combustion chambers of a diesel engine requires atomizing an abrasive slurry fuel with accurately sized orifices. Five injector orifice materials were evaluated: diamond compacts, chemical vapor deposited diamond tubes, thermally stabilized diamond, tungsten carbide with cobalt binder, and tungsten carbide with nickel binder with brazed and mechanically mounted orifice inserts. Nozzle bodies were fabricated of Armco 17-4 precipitation hardening stainless steel and Stellite 6B in order to withstand cyclic injection pressures and elevated temperatures. Based on a total of approximately 200 cylinder hours of engine operation with coal water mixture fuel, diamond compacts were chosen for the orifice material.

KEY WORDS

brazing, coal-fueled diesel, coal water mixture fuel, injection nozzles, diamond compacts, precipitation hardening stainless steel, active braze alloys, thermally stabilized diamond, CVD diamond, tungsten carbide/cobalt, tungsten carbide/nickel, Stellite 6B

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1. INTRODUCTION

Components of a coal-fueled diesel locomotive engine coming in contact with coal water slurry (CWS) fuel flowing at high velocities have to be orders of magnitude more erosion resistant than their conventionally fueled diesel engine counterparts. The orifices of the injection nozzle are particularly vulnerable to CWS wear. The CWS consists of finely ground coal, with a mean particle size of between 3 and 10 μm and ash content reduced to between 0.5 and 2%, mixed with water. At an injection pressure of approximately 82.7 MPa (12 kpsi), the resultant fuel velocities through the nozzle orifices are hundreds of meters per second. Because solid particle erosion is proportional to a power function of velocity (Menguturk and Sverdrup, 1979, p. 209), it is most severe at the nozzle orifices, where the velocity of the CWS is greatest. These orifices must maintain their nominal size (0.457-mm [0.018-in.] diameter) and geometry within just a few percent over the life of the nozzle in order to atomize the fuel efficiently at consistent rates so that the engine will operate smoothly and efficiently.

In earlier General Electric Corporate Research and Development work,¹ using continuous flow erosion tests at 27.6 MPa (4 kpsi) differential pressure, polycrystalline diamond compacts, and cubic boron nitride were identified as being sufficiently erosion resistant to be possible candidates for injector nozzle orifices (Mehan et al., 1991, p. 19). Since cubic boron nitride was not commercially available in properly sized disk form and diamond compacts, which were available, had the structural integrity to survive the stresses to which a locomotive engine injector nozzle would be subjected, diamond compacts were used for the prototype nozzle injectors for engine tests using CWS fuel. The material chosen for the injector body was 17-4 precipitation hardening stainless steel (PH S.S.).

2. NOZZLE DESIGN CONSIDERATIONS

A schematic drawing showing the mechanical design of the CWS fuel injector is shown in Figure 1. One injector is installed in each cylinder head with the nozzle protruding into the combustion chamber. A detailed drawing of the final brazed nozzle design is shown in Figure 2. Each nozzle consists of a housing, ten orifice inserts, and a conical valve seat.

¹ For DOE under contract DE-AC21-85MC22181.

The nozzle functions as follows: As the piston approaches "top dead center" on the compression stroke in any cylinder of the engine, the needle tip is raised 1 mm (0.04 in.) from the seat by the injector (Johnson and Hayden, 1994). The CWS fuel then flows to the nozzle orifices because the fuel pressure in the injector chamber is approximately 69 MPa (10 kpsi) higher than in the combustion chamber. The CWS flows rapidly into the sac and the holes in the housing that communicate with the orifices. The ratio of the flow areas must be large enough that most of the pressure drop and the highest velocities occur in the orifices so that the fuel is atomized as it enters the combustion chamber and wear can be controlled. Initial combustion studies in the GE Transportation Systems (GETS) single cylinder engine established the orifice hole diameter at 0.457 mm (0.0180 in.) and the number of orifices at ten.

Shortly before the required volume of fuel has been injected, the needle is accelerated toward the seat by large differential fluid pressure within the injector and begins to close. The objective closing time is 5.0×10^{-4} seconds, which requires a uniform acceleration of 830 g's. The valve seat in the nozzle must therefore be both impact and wear resistant.

In light of the results from earlier wear tests and engine tests with metal nozzles, it was decided to use diamond inserts for the orifices since the extrapolated life expectancy was thousands of hours (Mehan et al., 1991, p. 19). This material was especially attractive because compacts in the form of wire-drawing die blanks were available commercially and the technology existed for machining accurate holes through them to a tolerance of 2.54×10^{-3} mm (± 0.0001 in.).

Each diamond compact consists of a sintered cylindrical diamond core surrounded by a ring of cemented tungsten carbide (WC+Co) for support and to facilitate mounting (Figure 3). The GE Compax[®] diamond compacts, Blank No. 5235, that we used are 4 mm (0.157 in.) OD $\times$ 1.5 mm (0.059 in.) thick. In order to maintain the nozzle size within the required space envelope, these blanks were later ordered with OD reduced to 2.74 mm (0.108 in.)

The diamond inserts require a means of mounting and sealing that will reliably withstand the engine operating conditions: pressure alternating between 0 and 82.7 MPa (12 kpsi) approximately 9 times per second (at rated engine speed of 1050 rpm), nozzle housing temperature of

~191°C (375°F), and potential for hot corrosion. Brazing is commonly used to attach diamond die blanks.

The decision to use brazing established the following additional design considerations:

- Brazing temperature limited to 729°C (1345°F) to prevent sintered diamond degradation (Gilmore et al., 1985, p. 22). Time at temperature during the braze cycle limited for the same reason.
- Alloy choice driven by difficult-to-wet sintered tungsten-carbide-with-cobalt-binder (WC+Co) material surrounding the diamond on the commercial diamond die blanks.
- Joint strength designed for a static 207 MPa (30 kpsi) pressure at room temperature, 2.5 times the 0 to 82.7 MPa (0 to 12 kpsi) cycle pressure inside the nozzles. The 2.5-X design margin allows for fatigue and a ~191°C (375°F) operating temperature, based on temperature measured within a nozzle housing near the orifices.
- Fits between compacts and housing sized to allow for coefficient of expansion differences to maintain a strength-optimizing 0.051 mm (0.002-in.) braze gap at brazing temperature.

The nozzle housing must provide adequate structural support for the orifice inserts oriented nearly radially outward, a hard seat insert for the injector needle valve, leak-tight mounting to the injector lower housing, and adequate fatigue resistance for tens of millions of injection pressure cycles. It must also have good corrosion- and erosion-resistance and offer reasonable machinability. Its outside diameter must not exceed 24.8 mm (0.975 in.) where it penetrates the cylinder head.

A mechanical design concern, which has been an occasional source of failure in conventionally fueled diesels, is the stress across the ligaments between the holes through which fuel flows to the orifices. The calculated maximum tensile stress occurs across the ligament between holes at the surface of the sac.

3. NOZZLE DESIGNS AND EXPERIENCES

In the earlier development work,² 17-4 PH S.S. had been selected for the housing because it has a high fatigue limit (503 MPa [73 kpsi]) and it is strengthened and hardened by a simple heat treatment, achieving Rc43 in condition H 900 (Armco, 1986). Its fatigue limit is significantly higher than 276 MPa (40 ksi), the maximum tensile (hoop) stress in the body of the nozzle. It also has good corrosion resistance, weldability, and machinability; all comparable to austenitic stainless steels.

Initially, the diamond compacts were directly brazed into pockets machined into the housing (Figure 4). After two incidents in which orifice inserts were pushed out of the housings (further details are given in Section 4.1), an effort was made to hold the inserts in pockets mechanically by using retainers that were electron beam (EB) welded to the housing. Predrilled diamond compact inserts were mechanically mounted into individual 17-4 PH retainers that were then EB welded into the housing (Figure 4, Part 4A). This mounting technique was first incorporated in an 8-orifice nozzle that was run for approximately 80 hours in the GETS single-cylinder combustion engine. From prior experience with CWS, it was expected that the fuel particles would pack into and seal any crevices between the orifice inserts and the housing. This sealing did not take place in all instances, however. In one nozzle, sufficient material was eroded between inserts and housing that one insert rotated 90° in the pocket (shown in Figure 15). At that point, it was decided that inserts would have to be brazed into retainers that were subsequently EB welded into the housing. Unfortunately, the nozzles for the 12-cylinder GETS engine were irreversible committed to fabrication using the mechanical orifice retention.

Because some of the inserts exhibited severe loss of material at the interface between the tungsten carbide and the diamond, as shown in Figure 5, two 10-orifice nozzles (designated #16 and #19) were built and run in the GETS "durability engine" to evaluate potential alternative orifice materials. A total of three types of diamond inserts and two carbides were tested with both brazed and EB welded, and "mechanical" insert mounting. Identical orifice materials mounted

² For DOE under contract DE-AC21-85MC22181

identically were oriented at 180° in each nozzle. Fabrication and testing of these nozzles is described in Section 4.3.

The diamond nozzle inserts were fabricated from three types of polycrystalline diamond material. One type was a GE Compax diamond compact, Blank No. 5235, which is fine particle size polycrystalline diamond containing some metallic sintering aid produced in the core of sintered tungsten carbide cylinders at very high pressure and temperature. The tungsten carbide cylinder becomes a retaining ring for the sintered diamond core. Common practice to enhance the sintering of diamond is to infiltrate a liquid metal diamond-growth catalyst through the interstices of the diamond particles. For diamond compacts, the cobalt-alloy binder in the cemented carbide is the primary source of sintering aid. As a result, the diamond produced by the metallic-catalyst-enhanced sintering method contains some residual catalyst metal that is dispersed throughout. Because of the significant difference in thermal expansion characteristics of diamond and the residual catalyst, this material tends to develop cracks when heated above some critical temperature. The critical temperature varies, depending on the particle size of the diamond, the amount of residual catalyst, etc.; but, according to the recommendations of manufacturers of these materials, heating above 700°C (1292°F) can cause significant damage. In addition, diamond compacts sometimes show preexisting microstructure cracks that limit the range of potential applications.

As noted, the sintering of diamonds requires the infusion of liquid cobalt from the cemented carbide retaining ring. This process also results in the dissolution of the diamond into the liquid metal, however, which causes the liquid cobalt in the cemented carbide to become supersaturated with carbon. When the metal freezes at the end of the sintering process, this excess carbon in the metal can precipitate out as graphite particles in the cemented carbide binder phase, weakening the carbide structure near the diamond/carbide ring interface. This "carbon porosity" not only reduces the mechanical strength, it can also reduce the oxidation resistance of the structure. If the environmental conditions in the engine cylinder are conducive to oxidation, the carbide side of the interface region between the diamond core and the carbide jacket oxidizes faster than the normally expected rate for cemented carbide. Figure 5 is a photograph of the exit face of two orifices from the 8-orifice nozzle showing signs of significant degradation. The general

appearance of the degraded zone looks very much like a similar carbide part that was subjected to hot corrosion testing, but this observation was not verified with independent tests.

Another type of diamond material, thermally stabilized diamond (TSD), is commercially produced by following the initial steps for making diamond compacts but using a chemical process to selectively remove the original carbide jacket and most of the residual catalyst metal in the sintered body. Removing the residual metal from the composite improves the material's ability to withstand elevated temperatures, but tends to decrease its toughness compared with as-processed material.

Another type of diamond material, diamond tubes produced in the laboratory at GE-CRD by the chemical vapor deposition (CVD) process, was made by depositing diamond on surfaces of refractory metal wire substrates, then removing the substrate remaining at the core of the diamond layer by chemical means. Because the CVD process is used on the surface of a cylindrical substrate, the material produced tended to have elongated grains oriented radially. As a result, the microstructure has weak grain boundaries lined up normal to the axis of the tube, making the tube weak under axial tension.

Figures 6, 7, and 8 show the exit ends of three orifices. The orifice made from diamond compacts shows very little sign of wear, and the original geometry of the orifice outlet is very well maintained (Figure 6). The end of the orifice produced from TSD (metal removed) blanks has been fractured, although there are no indications of wear on the inside of the orifice (Figure 7). The orifice made of CVD diamond tube also shows a fractured end, even though it too shows no indication of wear (Figure 8) on the inside. The damaged surface of the orifice shows that it failed by intergranular fracture and that the grains were elongated, as noted previously, and the boundaries run through the thickness of the tube. The internal surface of the orifice also shows that the cross section of individual diamond grains is large and that the boundaries of the diamond grains have been eroded.

We concluded, from the results of durability engine tests of nozzle insert materials, that all the diamond-based materials have sufficient wear resistance to survive for high-pressure, high-velocity injection of coal slurry, but they are not all strong enough to survive the tensile stress applied to the orifice exit by the slurry. It seems that diamond compact with metal binder can

withstand the tensile stress, but the tensile strength of TSD compacts and CVD diamond tubes would have to be improved to make them usable. Also, the potential exists for hot corrosion damage of the diamond compacts.

Nozzle inserts were fabricated of tungsten carbide with nickel binder and tungsten carbide with cobalt binder. All carbide orifices demonstrated observable wear at the exit edge at zero or low magnification. We conclude, therefore, that cemented carbides will not be suitable orifice materials.

Nozzle seats and injector needle tips were originally made of tungsten carbide with 6% cobalt binder. The seats were shrink fitted into the nozzle bodies to induce compression in the seat in order to prevent cracking due to tip impact. After as little as 25 hours running time, pitting could be seen in the surfaces of both parts. A series of corrosion tests was conducted at GE-CRD in which samples of tungsten carbide with cobalt binder and tungsten carbide with nickel binder were exposed at room temperature to CWS from Otisca, Inc., and CQ, Inc. (Johnson and Hayden, 1994). Weight losses for the carbides with nickel binder were one-third those of cobalt carbides, and surface appearance was notably better. The durability test nozzles and all nozzles in the multi-cylinder engine had seats made of WC+10% nickel binder. (Ten percent binder content was selected rather than 6% as a conservative measure against structural failure since not enough time was available to run screening tests.) The seats and needle tips from the durability tests were examined after the testing was completed. Slight localized indentations were present, but no failures had occurred and no operational problems were reported. We conclude that cemented tungsten carbides with appropriate binder material are good candidates for valve-type applications in the nozzles.

In order to obviate stress across the ligaments between the holes through which the fuel flows to the orifices, a vertical offset between adjacent holes was incorporated to significantly increase ligament area. Slight rounding of the entrances of the holes, resulting from erosion, also reduced stress concentration and thereby helped to offset the increase in stress caused by material lost by this rounding. Including stress concentrations, the maximum tensile stress at the ligaments is 221 MPa (32 ksi).

4. ORIFICE INSERT JOINING DEVELOPMENT

4.1 BAg-3 Braze Assembly of Diamond Compacts in 17-4 PH S.S. Nozzle Housings

A torch brazing technique was developed (see the Appendix for details) to join diamond compacts into 17-4 PH housings using a conventional and commercially available silver braze alloy, 50%Ag/16%Cd/15.5%Cu/15.5%Zn/3%Ni (specified as BAg-3 in AWS A5.8; 632°C (1170°F) solidus/ 688°C (1270°F) liquidus). *Before* and *after* photographs of a brazed nozzle assembly are shown in Figure 9. This nozzle was engine tested using CWS fuel for over 50 hours with no apparent wear or braze joint failures. However, a *significant* reduction in hardness was measured in the 17-4 PH injector housing body as a result of overaging during the braze thermal cycle.

Three steps were taken in an attempt to resolve the problem with overaging of 17-4 PH during brazing. First, an injector housing was made of Stellite 6B and tested. However, Stellite 6B was dropped from further consideration because of its poor machinability. Next, time at temperature was halved (reduced from 10 minutes to 5) using the BAg-3 alloy with 17-4 PH. Finally, at the suggestion of personnel from GE Superabrasives (manufacturers of the compacts), a lower melting temperature silver braze alloy was used: BAg-1, 45%Ag/24%Cd/15%Cu/16%Zn. The BAg-1 alloy allowed a reduction in braze temperature from the 721°C (1330°F) BAg-3 braze temperature to 660°C (1220°F). Each change in material or braze technique was tested by making mock-up assemblies, as shown in Figure 10, which were statically pressurized at room temperature to 207 MPa (30 kpsi), pressure being applied to the back of the diamond compact through a 0.762 mm (0.030 in.) diameter hole in the 17-4 PH or Stellite 6B holders. All the tests were successful.

The result of these changes was that 3 of the 10 compacts in the Stellite 6 nozzle were pushed out of their counterbores and one compact in a 17-4 PH nozzle brazed with the BAg-1 alloy was pushed out after less than 50 hours of engine testing. Because of the delay in the braze joint failure and because a lower melting temperature braze alloy was used, creep was suspected as the failure mechanism. An injector nozzle was then instrumented to measure engine operating temperatures in the orifice region of the nozzle housing. The operating temperature measured was 191°C (375°F). The generic strength versus temperature curves for silver braze alloys shown

in Figure 11 support the theory of creep failure in an injector operating at 103 MPa (15 kpsi) maximum pressure and 191°C (375°F).

The reduced time at temperature and the change in braze alloy had another consequence, which was probably related. The WC+Co support section on most of the diamond compacts brazed with shorter hold times or with BAg-1 did not wet completely, unlike the first two 17-4 PH/diamond compact nozzles brazed with the technique described in the Appendix. The wetting problem made the joint gap between the WC+Co and nozzle housing counterbores susceptible to incomplete capillarity and fill. To prevent diamond degradation, we were already brazing at 32 to 60°C (90 to 140°F) below the recommended temperature for brazing of tungsten carbide with BAg-3, the recommended alloy for WC (*Brazing Book*, 1977, pp. 48-49) and with very short heat-up and hold times. The need to deal with the overaging as well made it unlikely that we could make silver braze joints of sufficient strength consistently, especially for use in a 191°C (375°F) operating environment. As a result, we decided to try securing diamond compacts in 17-4 PH housings by EB welding a cap over them to hold them in place mechanically.

4.2 Electron Beam Welded 17-4 PH S.S. Housing with Diamond Compact Orifices

The design used for mechanically fastening diamond compacts in 17-4 PH injector nozzle bodies is shown in Figure 12. Smaller diamond compacts, 2.74 mm (0.108 inch) OD, were used in the EB welded design to permit the inserts to be located at a smaller radius. Use of the smaller compacts also reduced the parasitic volume within the nozzle body. Mock-ups were made to set welding parameters and to check for possible thermal degradation of the diamond compacts. Parameters for EB welding were established for making sound welds of the required penetration, which was checked by metallographic examination of mock-up cross sections (Figure 13) and pressurization tests at room temperature. Each circumferential weld was completed in 0.3 second with an energy input of 350 joules. No thermal degradation of the diamond compacts occurred using these parameters. The only precaution taken to prevent diamond degradation was to set a minimum time between welds of 5 minutes to reduce heat buildup in the housing with successive welds and to restrict overaging of the 17-4 PH to the area adjacent to the welds.

Several nozzles were assembled by EB welding and engine tested. Before and after photographs of one of them are shown in Figure 14. After approximately 80 hours of engine

testing on Nozzle #4, several of the housing cavities and caps were eroded to the point where the diamond compacts were loose in the cavity (Figure 15). Visual examination led to the conclusion that coal slurry fuel was being forced through the unsealed gap between the diamond compacts and the 17-4 PH housing pockets causing erosion and eventual failure; mechanical mounting without metallurgical bonding was abandoned. Unfortunately, fabrication of the nozzles for the 12-cylinder engine was already committed.

4.3 Durability Nozzle Joining Development

4.3.1. Test Matrix

The cobalt matrix in the sintered diamond material on the combustion chamber faces of the GE Compax diamond compacts showed evidence of hot gas corrosion, as shown earlier in the photograph in Figure 5. As a result, the following materials were investigated as additional candidates for coal diesel nozzle injector inserts:

- Tungsten carbide with nickel binder (WC+Ni)
- Tungsten carbide with cobalt-chromium binder (WC+CoCr)
- Chemical vapor deposited (CVD) diamond tubes (made at GE-CRD)
- CVD diamond tubes coated with tungsten by GE Superabrasives
- Commercial thermally stabilized diamond compacts
- TSD diamond compacts coated with tungsten or with titanium carbide/nickel (W+TiC/Ni)

The tungsten carbide materials were chosen to test the possibility of using diamond compact binder materials other than cobalt, since it appeared to be the cobalt binder in the GE Compax diamond compacts that was being attacked. As noted in Section 3, in room temperature laboratory corrosion tests, weight losses for the carbides with nickel binder were one-third those of cobalt carbides. The TSD material is a commercially sintered product like the one used in diamond compacts, except that the cobalt matrix is removed after the press and sintering operation. The removal of the cobalt matrix prevents thermal degradation of the diamond-to-diamond bonds at elevated temperatures caused by the difference in coefficient of thermal expansion between the diamond particles and the cobalt matrix. Since CVD diamond tubes are produced by depositing diamond on surfaces of refractory wire substrates that are later removed

chemically, the need to machine orifice holes is eliminated. The tungsten coating on the CVD diamond and the tungsten and titanium carbide/nickel coating on the TSD diamond were investigated as means of enhancing braze alloy wetting on these difficult-to-wet materials.

Because of the cap erosion experienced with the EB welded nozzle design, it was decided to braze the orifice materials into the 17-4 PH caps before EB welding them into the nozzle housing. A schematic of the braze and EB weld design is shown in Figure 16. The erosion was perceived to be caused by the flow of CWS in the gap between the diamond compacts and EB welded caps. The expectation was that the erosion would be prevented by filling the gap with braze alloy. At the same time, EB welding of the brazed orifice/retainer subassemblies was used to prevent the possibility of creep failure and subsequent orifice ejection.

Since all braze joints would entail brazing 17-4 PH to tungsten carbide, or to tungsten, in the case of the coated diamond material, the first test mock-ups (shown earlier in Figure 10) were made using pure tungsten, WC+6%Ni, and WC+10%Ni. The braze alloy in this case was BAg-3 applied manually with a torch, with and without pretinning of the tungsten and WC. All other procedures were as described in the Appendix. Torch brazing could be used since there was no danger of thermal degradation of tungsten, WC+Ni, or any of the other orifice materials used.

Testing of the mock-ups consisted of subjecting the specimens to a cyclic pressure of 0 to 103 MPa (15 kpsi) through the 0.762 mm (0.030 in.) diameter hole in the 17-4 PH holder against the back of the brazed-in orifice insert, at a test temperature of 316°C (600°F). This conservatively high temperature was chosen before data on the instrumented engine test nozzle had been gathered. The results of mock-up tests at this temperature and pressure cycle were time-dependent failures, again indicative of creep. All insert materials pushed out of their holders within 1 hour of test time.

Microscopic evaluation of the failed mock-ups revealed complete joint fill and wetting of both the 17-4 PH holders and insert materials, with only minor voiding. The pretinned specimens appeared to have a thicker braze joint at the bottom of the counterbore between the 17-4 PH and the orifice insert, as evidenced by the thickness of the "plug and socket" push-outs (see Figure 17). Two mock-ups were sectioned and polished after brazing. One was a pretinned tungsten orifice insert brazed into a 17-4 PH holder which was machined and then EB welded into a 17-4 PH nozzle housing, Figure 18. The other, a WC+6%Ni insert brazed into 17-4 PH relying on

capillarity only to fill and wet the joint, Figure 19. The metallographic mounts also revealed complete joint fill with only minor voiding in both cases. In the pretinned specimen, the joint thickness between the counterbore bottom and tungsten orifice inserts was double the thickness of the radial joint.

Braze alloy strength calculations were made based on excluding the braze joint at the bottom of the counterbore and assuming 103 MPa (15 kpsi) maximum pressure over the entire diameter of the orifice inserts. Insert sizes were 2.29 mm in diameter $\times$ 1.52 mm (0.09 in. diameter $\times$ 0.06 in.) or 3.55 mm in diameter $\times$ 1.52 mm (0.14 in. diameter $\times$ 0.06 in.). The calculations predicted a mean shear stress of 34.5 MPa (5 ksi) for small diameter inserts and 52.4 MPa (7.6 ksi) for larger inserts. These values are 25 to 50% less than the reported static strength of 69 MPa (10 ksi) at 316°C (600°F). (See plots in Figure 11.) It is likely, therefore, that creep was the mode of failure. Since the orifice insert would be loaded from the other side in the combined braze and EB weld design, with the 17-4 PH cap supporting it and preventing creep (see Figure 2), these tests did not indicate imminent failure. Only an instantaneous leak through the braze joint would have indicated failure since the intended function of the braze joint in this design is to prevent slurry flow between the orifice insert and the 17-4 PH holder.

Next, thermally stabilized diamond, coated first with titanium-carbide and then with nickel, was brazed into 17-4 PH mock-ups for testing. The coated TSD diamond material was provided by GE Superabrasives. BA-3 alloy was again used. The only difference in brazing technique between the W and WC+Ni samples was that the coated TSD diamond inserts were not grit blasted, but were rubbed with fine steel wool and rinsed with acetone to clean them because the S.S. White Al₂O₃ grit blast would have caused the coating to spall off. Testing was done at 260°C (500°F) and 0 to 82.7 MPa (12 kpsi) pressure through the 0.762 mm (0.030 in.) diameter hole in the 17-4 PH holder. The result was that the TSD diamond insert was not pushed out, but almost immediately (within 30 seconds) began to leak through the joint. Microscopic examination of the exposed joint revealed that the 0.038 mm (0.0015 in.) thick (nom.) braze joint was still intact, bonded to the 17-4 PH. However, there was now a 0.0762 to 0.127 mm (0.003 to 0.005 in.) gap between the braze joint and the TSD diamond, around the entire circumference (see photograph in Figure 20). Since there was a correlation between the width of the gap and coating thickness, it was assumed that the coating had been displaced during testing. A SEM

Energy Dispersive X-ray Spectrometry (EDS) scan of the failed joint showed no indication of nickel, supporting the conclusion that the coating had been displaced. Since it was evident that the TiC/Ni coated TSD diamond material would not provide a leak tight joint, at least not under expected pressure/temperature conditions, it was dropped as a candidate material.

With the failure of the TiC/Ni coating, and indications (a white "soot" in the 0.457 mm (0.018 in.) hole in the orifice material) of vaporization of one or more of the constituents of the BAg-3 alloy during vacuum EB welding, consideration was given to choosing higher melting temperature braze alloys, without low vaporization elements such as cadmium and zinc, i.e., braze alloys that would wet carbides and diamond directly. Besides preventing vaporization and possible remelting of the braze alloy during EB welding, higher melting temperature alloys would reduce the likelihood of creep at engine operating temperatures, and therefore could permit the use of the braze-only design with the non-thermally degradable orifice materials. After consulting brazing references (*Brazing Book*, 1977, pp. 38-39, 46-50; *Brazing Manual*, 1963, pp. 223, 241; *Metals Handbook*, 1983, pp. 1001-1004, 1063), and phone conversations with Lucas-Milhaupt, Inc., and GTE WESGO, Inc., the six alloys shown in Table 1 were chosen for investigation.

Table 1. Alloys Chosen for Investigation

AWS spec. A5.8	Trade Name	Manufacturer	Composition (wt%)	Solidus/Liquidus °C/°C (°F/°F)	Insert Metal Brazed with Alloy
1 BAg-3	Easy-Flo 3	Lucas-Milhaupt	50Ag/16Cd/15.5Cu 15.5Zn/3Ni	632/688 (1170/1270)	WC+Ni
2 BAg-23	Braze 852	Lucas-Milhaupt	85Ag/15Mn	960/971 (1760/1780)	WC+Ni
3 BAu-4	Nioro	GTE WESGO	82Au/18Ni	949/949 (1740/1740)	WC+Ni
4 BNi-9	Nicrobraz 150	Wall Colmonoy	15Cr/3.5B/.06Cmax bal. Ni	1054/1054 (1930/1930)	WC+Ni
5	Incusil ABA	GTE WESGO	59Ag/27.25Cu/12.5In 1.25Ti	605/715 (1121/1319)	CVD Diamond and WC+Ni
6	Lucanex 721	Lucas-Milhaupt	67.7Ag/26.3Cu/6Ti (nom) (72Ag/28Cu)	779/779 (1435/1435)	TSD Diamond and WC+Ni

Incusil-ABA and Lucanex 721 alloys are recently developed "active" braze alloys that have been reported successful in wetting oxides, nitrides, carbides, and other non-metallic materials without prior metallization. Wetting is achieved by adding titanium, which activates the alloys to form metallurgical bonds by the formation of one or more titanium compounds, i.e., titanium-oxides, titanium-nitrides, and/or titanium-carbides. The ability of each of these alloys to wet diamond was tested. Two pieces of CVD diamond sheet material approximately 6.35 mm × 6.35 mm × 0.33 mm (0.25 in. × 0.25 in. × 0.013 in.) thick were cleaned by Al₂O₃ grit blasting followed by 5 minutes of ultrasonic agitation in acetone. A 6.35 mm × 6.35 mm × 0.051 mm (0.25 in. × 0.25 in. × 0.002 in.) thick foil of Incusil-ABA alloy was placed over each of the cleaned CVD sheets, one on the "shiny" side, the other on the "dull" side. The shiny side corresponds to the smooth surface of the sheet that was adjacent to the substrate on which the diamond was grown by CVD. The dull side was the rougher free surface. The samples were vacuum fired at 749°C (1380°F) for 20 minutes. The photograph in Figure 21 shows complete wetting on both the shiny and dull side samples. Figure 22 shows micrographs of cross sections of both samples. A metallurgical bond is apparent with alloy flow within the crevices of the rough-surfaced dull side specimen. The same flow test was performed using Lucanex 721 alloy on the dull side of a CVD sheet using alloy in paste form. Figure 23 shows how the paste alloy flowed from the center of the sheet to wet about 80% of the area after vacuum firing at 927°C (1700°F) for 20 minutes. The micrograph of Figure 24 shows good flow within crevices and metallurgical bonding with the diamond with this alloy as well. Finally, a mock-up assembly similar to that shown in the sketch of Figure 10 was prepared and vacuum brazed with Incusil-ABA paste alloy. Figure 25 shows the CVD orifice and macros of the assembly before and after brazing. After brazing, the top surface of the assembly was polished and photographed (Figure 26) to show the metallurgical bond with both the diamond and the 17-4 PH holder.

Because of limited human resources and time, it was decided to run an abbreviated test matrix of 12 specimens using the six braze alloys listed in Table 1 with only two of the orifice materials, WC+6%Ni and uncoated CVD diamond tubes. These orifice materials were chosen because they were the most likely candidates and, at the same time, similar enough to WC+10%Ni and TSD diamond to be considered representative. In the case of the WC+Ni, the only variable was the alloy used, which accounted for half of the test matrix. The other six

specimens were CVD diamond brazed with Incusil-ABA and Lucanex 721 alloys using various forms of alloy and placement methods, and various joint gaps. Details are listed in Table 2. These variables were made to test effects on capillarity and resultant joint strength. Pre-braze cleaning involved Al_2O_3 grit blasting of the WC+Ni, CVD diamond, and 17-4 PH holders followed by 5 minutes of ultrasonic agitation in acetone. Foil and wire alloy was manually abraded with Al_2O_3 sandpaper and wiped with acetone. The test specimen geometry was the same as shown in Figure 10. Other pertinent details are included in Table 2.

A test schedule was devised with two goals in mind. First, to test for survivability under expected operating conditions with the "braze and EB weld design" in mind. That is, could the specimen hold 0 to 103 MPa (15 kpsi) pressure against the exposed face of the orifice insert for an extended period of time at 191°C (375°F)? If the specimen met this primary criterion, it would then be reversed and tested for potential use in a "braze only design," since the higher-melting-temperature alloys should be more creep resistant. Testing was done incrementally in order to establish failure mode, and to rank the materials. The sequence of testing steps follows:

1. At room temperature, gradually increase the static pressure, without cycling, on the exposed face of the insert material (WC or diamond) to the expected maximum injector operating pressure of 103 MPa (15 kpsi). Hold for 15 minutes, testing for leakage. These conditions preclude the possibility of creep, fatigue, or elevated temperature strength reduction.
2. At the expected operating temperature of 191°C (375°F), cycle the pressure on the exposed face of the insert for one hour to test for "braze and EB weld design" survivability under simulated engine service conditions.
3. Repeat Step 1 to test for leak tightness.
4. At room temperature, gradually increase the pressure on the back of the insert (through the 0.030 in. hole in the 17-4 PH and against the bottom of the counterbore of the insert (in a potential "push-out" mode) up to 103 MPa (15 kpsi). Hold for 15 minutes, testing for leakage. If the insert is pushed out, make note of the pressure.

**Table 2. Test Matrix of WC+6%Ni and CVD Diamond Tubes Brazed
with High Temperature and Active Alloys**

I.D. #	Orifice Material	Braze Alloy	Braze Process	Joint Gap*	Remarks
WC-1	WC+6%Ni	BNi-9	Vacuum; 1099°C (2010°F)/15 min	0.038mm (0.0015")	WC dipped in paste alloy, inserted, paste added on top
WC-2	WC+6%Ni	BAG-3	Manual Torch w/FB3A Flux	0.051mm (0.002")	0.002"-thick foil on bottom, 1/32"-dia. wire placed on top by hand
WC-3	WC+6%Ni	BAu-4	Vacuum; 1021°C (1870°F)/15 min	0.051mm (0.002")	0.002" foil on bottom, 0.082 gm of 0.040" dia. wire placed on top
WC-4	WC+6%Ni	BAG-23	Vacuum; 1021°C (1870°F)/15 min	0.051mm (0.002")	0.073 gm of 1/16"-dia. wire placed on top
WC-5	WC+6%Ni	Incusil-ABA	Vacuum; 749°C (1380°F)/15 min	0.064mm (0.0025")	0.002" foil on bottom, WC dipped in paste, inserted, paste added on top
WC-6	WC+6%Ni	Lucanex 721	Vacuum; 910°C (1670°F)/20 min	0.064mm (0.0025")	WC dipped in paste alloy, inserted, paste added on top
CVD-1	CVD diamond	Incusil-ABA	Vacuum; 749°C (1380°F)/15 min	0.038mm (0.0015")	diamond dipped in paste alloy, inserted, paste added on top
CVD-2	CVD diamond	Incusil-ABA	Vacuum; 749°C (1380°F)/15 min	0.038mm (0.0015")	0.002" foil on bottom, diamond dipped in paste, paste added on top
CVD-3	CVD diamond	Incusil-ABA	Vacuum; 749°C (1380°F)/15 min	0.076mm (0.003")	two 0.002" foils on bottom, diamond dipped in paste, paste added on top
CVD-4	CVD diamond	Lucanex 721	Vacuum; 910°C (1670°F)/20 min	0.076mm (0.003")	diamond dipped in paste, inserted, paste added on top
CVD-5	CVD diamond	Lucanex 721	Vacuum; 910°C (1670°F)/20 min	0.038mm (0.0015")	diamond dipped in paste, inserted, paste added on top
CVD-6	CVD diamond	Lucanex 721	Vacuum; 910°C (1670°F)/20 min	0.038mm (0.0015")	paste added on top only to see how well alloy capillared

* *At room temperature.* Considering mean coefficients of expansion of 5.4×10^{-6} cm/cm°C (3×10^{-6} in./in.°F) for WC, 10.8×10^{-6} cm/cm°C (6×10^{-6} in./in.°F) for 17-4 PH S.S., and zero for diamond, and a 2.54 mm (0.1 in.) nominal orifice insert diameter, radial joint gaps at braze temperature would be about 0.0051 to 0.0076 mm (0.0002 to 0.0003 in.) larger in the case of WC, and 0.0102 to 0.0152 mm (0.0004 to 0.0006 in.) larger for diamond, depending on the braze temperatures listed in the table.

5. To check for creep before there is any possibility of fatigue, gradually increase static pressure to the back of the insert to 103 MPa (15 kpsi) at 191°C (375°F). Hold for 15 minutes. Then check to see whether there was any movement of the insert. If the insert is pushed out completely, note pressure and elapsed time.
6. If the insert shows no creep during Step 5, cycle the pressure on the back of the insert between 0 and 103 MPa (15 kpsi) at 191°C (375°F) for 1 hour to test "brazing only design" survivability.
7. At 191°C (375°F), gradually increase static pressure on the back of the insert to maximum test stand pressure (~152 MPa [22 kpsi]) and hold for 10 minutes in an attempt to rank the alloys that survive all the preceding test steps.

4.3.2. Test Results

All twelve test specimens survived the first six steps of the test sequence. Only two failed during Step 7: WC-4 failed at 138 MPa (20 kpsi) and WC-6 failed at 152 MPa (22 kpsi).

To gain more information about the creep characteristics of WC brazed into 17-4 PH with BAg-3, specimen WC-2 was subjected to one additional test step. The pressure on the back was cycled between 0 and 103 MPa (15 ksi) at temperatures above 191°C (375°F). The test temperature was increased in increments of 27.8°C (50°F) and held for 15 minutes at each test temperature. The WC insert survived 15 minutes at 218°C (425°F), but pushed out after approximately 3 minutes at 246°C (475°F). Immediately after this test, the GE-CRD test stand had to be decommissioned. Had time allowed, it would have been beneficial for alloy ranking to subject the other 9 unfailed specimens to this test.

The test results indicate that any of the six alloys could be used successfully to braze WC+6%Ni (and probably WC+10%Ni) into 17-4 PH holders for EB welding into nozzle injector housings. Likewise, Incusil-ABA and Lucanex 721 alloys could be used for brazing CVD diamond tubes (and probably TSD diamond) into 17-4 PH holders. Indications are also good for being able to braze WC and diamond directly into 17-4 PH nozzle bodies without EB welding. Since the BAg-3 alloy failed at just 55.6°C (100°F) above measured operating temperature, and the BAg-23 and Lucanex 721 alloys failed at 30 to 50% above maximum operating pressure,

these alloys may not provide a suitable margin of safety for direct brazing. Local variations in temperatures and unusually high pressures are probably to be expected and would have to be taken into account in future designs. More testing (to failure) would have to be done at elevated temperatures and pressures with the other three alloys and WC, and with diamond brazed with the Incusil-ABA and Lucanex 721 alloys, to determine the safety margin in a direct braze design.

The failure of brazed (BAG-3) WC+6%Ni at 55.6°C (100°F) above operating temperature supports the theory that creep was the failure mechanism responsible for the four orifice inserts being pushed out. BAG-1, the braze alloy used in the direct brazing of WC+Co to Stellite 6B and 17-4 PH has a lower melting temperature than BAG-3 and would therefore be expected to push out at an even lower temperature.

4.4 Multi-Material Nozzle Assemblies

Two durability test nozzles, Nozzles #16 and #19, were fabricated to investigate potential new orifice materials. Two inserts of each material listed in Table 3 (Nozzle #16) and Table 4 (Nozzle #19) were assembled, 180° apart, into 17-4 PH housings using the mechanical EB weld design (Figure 12), or the braze and EB weld design (Figure 16) as noted in the tables.

Table 3. Nozzle #16 Orifice Material and Mounting

Insert #	Orifice Material	Mounting
1,6	CVD diamond tube, tungsten coated*	Brazed and EB welded
2,7	GE Compax diamond compact, blank #5235	Mechanical (EB weld only)
3,8	CVD diamond tube (w/o W coating)	Mechanical (EB weld only)
4,9	Thermally Stabilized Diamond, #5015, W coated	Brazed and EB welded
5,10	GE Compax diamond compact, blank #5235	Brazed and EB welded

* Tungsten coatings were applied to CVD diamond and TSD inserts by GE Superabrasives to improve brazability with low-temperature braze alloys.

Table 4. Nozzle #19 Orifice Material and Mounting

Insert #	Orifice Material	Mounting
1,6	Tungsten Carbide (WC) with 6% Ni Binder	Brazed and EB welded
2,7	Thermally Stabilized Diamond, #5015 (w/o W coating)	Brazed and EB welded
3,8	WC with Cr-Co binder	Brazed and EB welded
4,9	GE Compax diamond compact, blank #5235	Brazed and EB welded
5,10	WC with Ni-Mo binder	Brazed and EB welded

All insert materials were manually torch-brazed with BAg-3 alloy using the same technique as sample #WC-2 (Table 2), except for the TSD insert in Nozzle #19. This insert had no tungsten coating and was brazed with Incusil ABA alloy in a vacuum furnace at 810°C (1515°F) for 20 minutes using sample #CVD-1 technique (Table 2).

Both nozzles were run in the durability test engine at GETS for approximately 50 hours. The discharge side of three diamond orifice inserts is shown in Figures 6 through 8. The diamond compacts exhibit the least evidence of material removal, and there is no evidence of attack at the interface between the carbide and diamond that was observed in the 8-orifice nozzle run on the GETS combustion test engine (See Section 3). As may be seen in Figure 27, there was significant removal of material from the metal support ring adjacent to the discharge of some orifices. There appears to be a correlation between orifice exit edge wear and retaining ring damage. This probably indicates that the spray cone angle is increased as a result of orifice wear.

4.5 Conclusions

Assembly of diamond compact orifices into 17-4 PH S.S. nozzle bodies for coal diesel nozzles was possible by direct brazing using silver-based braze alloys. However, because of the thermal sensitivity of the diamond and precipitation hardening 17-4, braze temperatures were limited to 729°C with hold times as short as five minutes. The use of braze alloys with melting points lower than 729°C (BAg-3 and BAg-1) resulted in occasional creep failures at engine operating temperature. Brazes made with the BAg-1 alloy, which has the lower melting temperature, were more prone to creep than brazes with BAg-3. Marginal superheat and short hold times resulted in inconsistent wetting of the difficult-to-wet tungsten carbide outer layer of the compacts.

As a result of the inconsistent wetting and the possibility of creep failure in service, nozzle assembly was attempted by EB welding retaining rings over diamond compact orifices. The open gap between the compacts and nozzle body were subject to erosion by the coal water slurry. This problem was solved by a combination of brazing and EB welding in which compacts were brazed into 17-4 inserts which were then EB welded into nozzle bodies. This technique proved to be successful for joining diamond compact orifices into 17-4 nozzle bodies by preventing the erosion and inhibiting creep.

Hot gas erosion of the cobalt bearing diamond compacts made it necessary to investigate the use of alternate orifice materials. The alternate materials; CVD diamond, thermally stabilized diamond, tungsten carbide + CoCr, and tungsten carbide + Ni; were less heat sensitive than diamond compacts allowing investigation of higher melting temperature braze alloys. Six different braze alloys were investigated for joining tungsten carbide + 6% Ni and two active alloys — Lucanex 721 and Incusil-ABA alloys — were tried on CVD diamond. The results were that all of the alloy material combinations tested successfully under conditions expected in the braze + EB weld design configuration. Test results also indicated that creep resistant direct braze joints could be made on WC+Ni with Nicro, Nicrobraz 150, and Incusil-ABA alloys, and on CVD diamond with Lucanex 721 and Incusil-ABA alloys. Engine life tests would be recommended however to confirm that conclusion.

5. RECOMMENDATIONS

1. Evaluate the use of nickel as the binder material in the WC ring of diamond compacts (According to Dr. H. Marek of GE Superabrasives, this is feasible).
2. Further develop direct brazing of inserts (one nozzle so assembled ran for approximately 50 hours without incident).
3. Optimize the nozzle geometry and material selection to achieve acceptable levels of structural erosion.
4. Conduct engine tests, with periodic examinations, for at least 500 hours.

6. ACKNOWLEDGMENTS

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APPENDIX. Brazing Procedures for Direct Brazing of Diamond Compacts into 17-4 PH S.S. Nozzle Bodies

Cleaning: Diamond Compacts and 17-4 PH S.S. were Al_2O_3 grit blasted with S.S. White Abrasive Unit then ultrasonically agitated in acetone for 5 minutes to remove grit. BAg-3 filler metal (0.002" thick foil and 1/32" diameter wire) was manually abraded with Al_2O_3 sandpaper then ultrasonically agitated in acetone for 5 minutes.

Assembly: Microbraz Green Stop-Off (Wall Colmonoy Corp.) was painted on the inside diameter of the 0.030" holes in the nozzle body leading to the counterbores to prevent plugging with braze alloy (except first nozzle, whose 0.030" holes did plug). Foil alloy was cut into a circular disk to fit within counterbores on the 17-4 nozzle body. Wire alloy was wound as a coil onto a mandrel and cut into rings whose diameter was approximately the same outside diameter as the diamond compact orifice inserts. The counterbores in the 17-4 were fluxed with AWS type FB3A paste flux. The braze alloy foil disks were then fluxed and inserted into the bottom of each counterbore. Next, each diamond compact was fluxed and inserted into the counterbores, chamfer side toward the alloy disk and counterbore bottom. The compacts were slightly chamfered to prevent interference with the counterbore corner radius and assure full contact of the compact face with the counterbore bottom and alloy disk. The diamond compacts were then either staked into place with prick punches every 90 degrees, or held in place with 0.016" diameter nickel wire, resistance welded to the 17-4 over the outer edge of the compact. The 1/32" diameter ring of braze alloy was then fluxed and set in place over the diamond compact. The alloy ring was held in place by resistance welding a 1/8" x 5/16" x 0.008" thick strip of nickel foil over each counterbore pocket. More paste flux was placed over each assembled counterbore pocket and allowed to dry. See sketch in Figure A-1 for cross-sectional view of assembled counterbore pockets.

Brazing: The prepared nozzle assembly was loaded into a quartz tube nose down as shown in Figure A-2 (except the first nozzle). The nose-down orientation was used to prevent gravity feed of the molten alloy into the 0.030" holes in the nozzle body. A type S thermocouple was placed within the inside cavity of the nozzle body. The loaded quartz tube was purged with argon for at least two hours. The quartz tube was then placed in a tube furnace preset at some temperature above the liquidus of the braze alloy, 720°C (1330°F) in the case of BAg-3 alloy. The nozzle body was heated to the preset braze temperature and held for some allotted time (usually 10 minutes). The quartz tube was then removed from the tube furnace and the nozzle was allowed to cool under flowing argon. A representative strip chart recording of the braze cycle is shown in Figure A-3. A photograph of a nozzle after brazing is shown in Figure A-4.

Figure 1. Coal water mixture fuel injector

Figure 2. Final nozzle design

Figure 3. Normal and magnified views of diamond compact

Figure 4. Directly brazed nozzle design; diamond compacts brazed into 17-4 PH

74589A1B

30x

74589A1F 30x

Figure 5. Exit face of orifices of 8-hole nozzle showing significant degradation

Figure 6. Diamond compacts orifice showing little wear and well-maintained original geometry of nozzle outlet

Figure 7. TSD orifice fractured, no indication of wear on the inside

**Figure 8. CVD diamond tube orifice with fractured end,
no indication of wear on the inside**

Figure 9. Diamond compacts in 17-4 PH nozzle housings before (left) and after (right) brazing

Figure 10. Braze mock-up assembly

Figure 11. Strength vs. temperature curves for silver braze alloys

Figure 12. Mechanical fastening design

Figure 13. Mechanical fastening mock-up showing EB weld penetration

Figure 14. Diamond compacts in 17-4 PH nozzle housings before (left) and after (right) EB welding

Figure 15. Diamond compact rotated out of position within eroded nozzle body pocket

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Figure 16. Braze and EB weld design

Figure 17. Tinned mock-ups after failure, showing large plug and socket push-out within thick braze joint

Figure 18. Section through pretinned tungsten orifice insert brazed into 17-4 PH holder

Figure 19. Section through WC+6%Ni insert brazed into 17-4 PH, relying wholly on capillarity to fill and wet joint

Figure 20. TSD and TIC+Ni coating after brazing and testing at 260° C, 0 to 82.7 MPa, showing gap caused by displaced coating

Figure 21. Results of wetting test with Incusil-ABA alloy on CVD diamond

Figure 22. Cross-sectional micrographs of specimens of Figure 21, showing metallurgical bond and good crevice fill

Figure 23. Results of wetting test with Lucanex 721 alloy on CVD diamond

Figure 24. Cross-sectional micrograph of specimen of Figure 23, showing metallurgical bond and good crevice fill

Figure 25. CVD to 17-4 PH mockup assembly before and after brazing with Incusil-ABA paste alloy

Figure 26. Top of CVD to 17-4 PH mock-up, polished after brazing, showing metallurgical bond with Incusil-ABA to both materials

Figure 27. Durability nozzles after testing, showing support ring wear

Figure A-1. Alloy location and orientation during braze cycle

Figure A-2. Nozzle assembly loaded into quartz tube

Figure A-3. Representative strip chart recording of braze cycle

Figure A-4. Nozzle after brazing, before final machining

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