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DEVELOPMENT OF TESTING AND ANALYSIS METHODOLOGY TO ASSESS THE LONG TERM DURABILITY OF POLYMERIC COMPOSITES AT HIGH TEMPERATURES

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A White Paper and Summary of a Workshop entitled

DEVELOPMENT OF TESTING AND ANALYSIS METHODOLOGY TO ASSESS THE LONG TERM DURABILITY OF POLYMERIC COMPOSITES AT HIGH TEMPERATURES

by

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INTRODUCTION

Feasibility studies, supported by NASA, were conducted at Boeing Commercial Aircraft Company and Douglas Aircraft Company to determine if a High Speed Civil Transport (HSCT) could be an economical vehicle for the early 21st century time-frame. The Boeing study focused on a Mach 2.3 design while the Douglas study focused on a Mach 3.2 design. The design guidelines were for a range of 5000-6500 nautical miles, a payload of 250-300 passengers, a 60,000 hour life, and a standard fare. Both companies concluded that the planes were viable concepts and that there would be a market for such aircraft in the next decade. The studies further showed that a Mach number higher than 2.3 could have economical operational advantages. However, these higher Mach numbers might require new materials and certification procedures. In order to meet the desired weight, range, and speed in an economical fashion, polymeric matrix composites (PMC) may have to be widely used. The average surface temperature of the structure on the Mach 2.3 design is approximately 330^{o}F while the average surface temperature on the Mach 3.2 design is approximately 550^{o}F. The 330^{o}F temperature is within the operational range of many of today's PMCs. However, the 550^{o}F temperature is above the certified operational temperature of today's polymer matrix composites. Thus, polymeric matrix materials must be developed which can operate at higher temperatures if a Mach 3.2 design is to use PMCs. For either design, the ability of the composite material to endure 60,000 flight hours in an operational environment, maintaining structural integrity, must be ascertained.

A workshop was held at the NASA Langley Research Center on March 14-15, 1990, to help assess the state-of-the-art in evaluating the long term durability of PMCs and to recommend future activities. Forty engineers and scientists with experience in the areas of design and evaluation of PMCs at elevated temperatures were invited to participate. A collection of the workshop presentations and list of attendees is available from the author. The purpose of this White Paper is to briefly summarize the workshop presentations, the findings of the workshop sessions, and to outline the future plans of the Materials Division of NASA Langley.

WORKSHOP SUMMARY

PRESENTATIONS

The presentations were divided into four sessions: Background, Structures, Testing and Analysis, and Material Development and Evaluations. Each session will be reviewed separately.

Background

Paul Hergenrother of NASA Langley gave a review of the current state of high temperature polymers. Paul stated that currently no polymer system, suitable as a composite matrix, will maintain chemical stability for 60,000 hours at temperatures above 525^{O}F even under a no load environment. New high temperature polymer systems are currently being developed and certified. However, Paul thinks that the development of a polymer system that will be chemically stable above 600^{O}F is not very likely. Regardless of the type of polymer developed, a composite matrix development program should include the following elements: (1) polymer screening to select candidates; (2) polymer optimization to obtain best overall combination of properties; (3) polymer scale-up; (4) prepreg development; (5) composite fabrication development; (6) thermal-mechanical testing including fatigue and creep of quality composites; (7) time/temperature/stress/humidity/etc. testing; and finally (8) prototype fabrication and testing. A program of this dimension must be done and will cost tens of millions of dollars and require approximately ten years to complete. Paul outlined several possible directions to develop suitable new polymers, such as a PMR-15 plus a thermoplastic blend.

Jim Haskins of San Diego State (formally with General Dynamics/ Convair Division) reviewed a large 1970's NASA sponsored program entitled "Time-Temperature-Stress Capabilities of Composite Materials for Advanced Supersonic Technology Application" (NASA Contractor Report 178272, May 1987). This was a long term (50,000 hrs) testing program involving a variety of then available potential high temperature composites. Test conditions included both a real-time service environment, and thermal and environmental aging. All of the polymeric based composites suffered from significant thermal oxidation resulting in a loss of mechanical properties. All of these systems had use temperatures, as established by the long term tests, that were considerably less than initially expected. The long term degradation of the matrix material was more significant in specimens tested with compressive loads. Many problems occurred during the course of the test program including equipment failures (pumps, recorders, valves, etc.), demise of composite systems (i.e. material no longer available), personnel changes, batch to batch variation of the materials, power failures, etc. Long term testing can be very taxing on men and equipment.

Sam Dastin of Grumman described an Air Force sponsored program entitled "Environmental Sensitivity of Advanced Composites" (AFWAL-TR-80-3076 Vol. 1, August 1979). The first objective of this program was to define a realistic environmental and loading spectra for an Air Force aircraft. The environment and loading spectra of the B-1 horizontal stabilizer was selected. The stabilizer design was a Gr/Ep substructure, 6 to 32 plies thick, with operating temperatures up to 260°F. They developed a Baseline and a Worst Case flight profile and conducted long term tests to 6 aircraft lifetimes. They found that residual tension strength was not affected by fatigue or environment but compression properties were reduced. They reported no significant synergistic effects of fatigue loading and environment.

Joe Storr of the Wright Research and Development Center reviewed a new USAF program to investigate Ultra High Temperature Organic Matrix composites. The program will encompass materials and component testing at 600-800°F. The funding and duration of the program will be over 3 years. The program is scheduled to start Fall 1990 with Kevin Boyd, WRDC/FIBEC, as the technical monitor.

Structures

Cliff Kam of Douglas presented an overview on the Douglas HSCT, specifically concentrating on the material durability and damage tolerance requirements. He suggested that creep may have to be included in the design and this would be a new consideration for airframe designers. He further suggested that creep strain be limited to 0.001 for one application of limit design load. He emphasized the need to do real time coupon, element, and structural testing. This testing could take 10 to 18 years, depending on the number of hours per day tests were conducted. Douglas is currently considering a wide variety of materials for their Mach 3.2 design, including aluminum matrix composites, high temperature aluminums, titaniums, and PMCs.

Glenn Grimes of Lockheed outlined a detailed "Building Block" approach for structural development. This approach builds from testing coupons, structural elements, and full scale structural components for certification. This building block approach was reiterated and endorsed by others from industry that were present. Glenn summarized by saying that a linear elastic composite material with predictable wearout and aging characteristics at 450°F could be accommodated into design with the current methodology. Linear/ nonlinear elastic composite materials would need new design methodology development, which may take ten years. Composite materials with pronounced viscoelastic behavior would require more difficult methodology development and may take over ten years.

Charlie Saff of McDonnell Aircraft Company gave an overview of structural certification. Charlie echoed many of the thoughts of Glenn Grimes, as to the building block approach and difficulties of designing and certifying viscoelastic structures. Further difficulties are expected with material oxidation and thermomechanical loads.

Robin Whitehead of Northrop shared some of his thoughts on durability of composite structures. Once again the building block approach was recommended. Robin reviewed an USAF sponsored program for designing a composite fighter wing/fuselage that saw a maximum service temperature of 250^{o}F. The composite laminate was cycled under a variety of temperature and load profiles, accelerated and real-time. Outstanding composite durability was demonstrated under extreme fatigue loading and environmental conditions. He concluded that no fatigue tests were needed for an all-composite structure and that static strength tests were adequate to identify structural hot spots. Robin described a materials use envelope in three areas as a function of moisture content, temperature and design strain: Reversible region - Essentially linear-elastic, no significant durability effects, all test schemes equivalent, and all failure modes have equivalent durability response; Transition region - Nonlinear/time-dependent effects, durability failures can occur, distinct differences between accelerated test schemes, real time testing may be needed, and testing certification requirements unacceptable; TG exceeded -Material essentially useless. In closing, Robin recommended for HSCT design to used material in the "reversible region" by making a smart material selection and using realistic design strain levels in fiber dominated lay-ups.

Testing and Analysis

Doug Ward of GE Aircraft Engine Division offered a detailed overview of some of the environmental tests used for screening and analyzing new materials. Examples were given for a PMR-15 matrix composite which showed severe matrix cracking under thermal cycling. William Wallis and Bob Boscham of Lockheed also suggested testing approaches for new resin systems. Bjorn Backman of Boeing Commercial also gave a review of desired materials properties and testing techniques. The critical specimens appeared to be hot-wet open hole compression and compression after impact. Bjorn pointed out that fiber waviness will become a more critical issue under compressive loading at higher temperatures because of the decrease in matrix properties.

Kyle Owen of General Dynamics/Fort Worth Division presented IRAD results for certification of some F-16 PMC components. General Dynamics is also using the building block approach for design. Their testing considerations for environmental characterization included thermal spiking, thermal cycling and time at temperature.

Wolfgang Knauss of Cal Tech spoke on the analytical relationships between material properties, such as free volume and creep, specific volume and temperature, and how to relate some of these properties to composite behavior. Hal Brinson of the University of Texas at

San Antonio also gave some approaches to lifetime predictions using a time-temperature-stress superposition (TTSSP) methodology. Hal presented various examples of creep and stress rupture predictions which showed that the current TTSSP methodology was adequate for predicting time dependent memory deformations.

Material Development and Evaluation

Mark Rogalski of Boeing described an approach for evaluating new PMC materials by trying to isolate the effects of moisture, thermal cycles, loads, and UV. This approach would aid in development of a lifetime predictive model. He stated that Boeing feels that the best speed for environmental/economic viability is Mach 2 to 2.5.

Rudy Gonzalez of Northrop B-2 Division reviewed an evaluation study of new processible polyimides. Most of the emphasis of the evaluation was on thermo-oxidative stability. They developed a high temperature organic matrix resin that demonstrated thermo-oxidative stability at 700°F for 100 hrs. This 100 hrs is far short of the 60,000 hrs needed for the HSCT.

Sam Boszar of Pratt & Whitney showed some results from thermal oxidative stability tests on PMR-15. He also looked at some effects of erosion by road dust that could be sucked into an engine. Several non-destructive evaluation techniques were discussed. A dielectric monitor was identified as a desirable NDE tool because of its sensitivity to oxidation, cracks, and chemistry. Ray Adsit of Rohr Industries also presented thermal exposure data on PMR-15, pointing out micro-damage and its adverse effect on several mechanical properties. Ken Bowles of NASA-Lewis presented the effect of different reinforcements on the thermo-oxidative stability of PMR-15.

Bob Buyny, Hexcel Corporation, compared the durability of PMR-15 and toughened BMI composites subjected to combined aging and thermal cycling. T300/HX1539 (a toughened BMI) exhibited one fourth the thermo-oxidative stability of C3000/PMR-15 at 500°F: however, the lifetime of the toughened BMI was 90% of the C3000/PMR-15 because the BMI experienced only minor surface cracking.

Summary of Key Observations

The workshop presentations may be summarized by the following key observations:

o Polymer chemists are not very optimistic about developing a resin matrix material that can exceed 600°F for 60,000 hrs.

o Previous long term durability testing programs were very expensive and extremely taxing to both personnel and equipment.

- The composite design community feels as if time-dependent/non-linear material properties should be avoided by smart material selection and/or lessening design requirements, otherwise a very difficult, long-term, and expensive methodology development program will be needed.

- Compression after impact and open hole compression tests are the critical tests for ranking materials in the current airframe design community.

- All airframe companies employ a building block approach for composite structural design and material certification.

- Thermal-oxidative stability appears to be the key test used to evaluate the high temperature capability of resin matrix composites.

WORKSHOP SESSIONS

The attendees divided into three working groups. Each working group was assigned the task of answering one of the following questions:

Working Group 1:

- What fundamental properties are important for materials evaluation and how should they be determined?

Working Group 2:

- How does one assess long term durability and develop appropriate accelerated test techniques?

Working Group 3:

- How does one assess the time dependent effects of a polymeric matrix composite used at high temperatures? What role does thermal aging play and how can it be modeled?

Each working session met for approximately two hours then reported their findings to the whole workshop. These findings are summarized in the following sections.

Working Group 1

Question: What fundamental properties are important for materials evaluation and how should they be determined?

This group was Chaired by Glenn Grimes of Lockheed, Rod Martin of Analytical Services and Materials (NASA Langley) served as the recorder, and five others were in attendance.

The discussion of the fundamental properties important for materials evaluation was split into two sections, static properties and time dependent properties. The static properties after short term temperature exposure at several elevated temperatures need to be determined. The static properties assessment should also include material aging to determine the response to time at temperature. The time dependent properties related to creep and fatigue testing issues must be evaluated in both real time and accelerated tests.

A list of mechanical property tests which are typically used for design was drawn up. Tests on the list fell into two categories, tests at the laminate level (e.g. quasi-isotropic) and tests at the lamina level. At the laminate level, the tests considered important for determination of strength included compression after impact (CAI), plain and open hole compression, tension, shear, bolt hole bearing, and interlaminar fracture toughness. Stiffness data should be obtained from plain compression, tension, and bearing tests. At the lamina level, strength and stiffness data should be obtained from 0° compression and tension, 90° compression and tension, and $\pm 45^{\circ}$ tension tests. All these tests should be conducted statically at elevated temperatures. Selected tests were chosen as the key properties to determine the effects of aging on strength and stiffness. These were CAI, plain and open hole compression and tension, and bearing. The effects of aging should be characterized by determining the weight loss, microcracking, stiffness degradation, and chemical changes (FTIR, Infrared spectroscopy). The aging tests should be conducted at a temperature slightly above the operational temperature to evaluate accelerated aging. Checks to ensure the failure mechanisms were not altered must also be conducted.

From the above tests, selected ones were chosen for the initial screening of the material. These tests were chosen to be the most critical ones. If the material does not meet the necessary requirements of these tests then it should not be considered a candidate material. It was decided that screening of each material should include short time tests and aging tests which take no longer than 6 months (approximately 4,000 hours) and should be done only at the coupon level. The tests chosen were short time and aged strength data from CAI, plain and open hole compression, 0° compression, and $\pm 45^{\circ}$ tension, and stiffness data from CAI, plain compression, 0° compression, and $\pm 45^{\circ}$ tension. The short time tests should be done at several temperatures and the aging tests at one temperature (e.g. operational temperature) at several times. It was considered possible to reduce the number of tests (e.g. omit plain compression) in the data base as experience is gained in identifying the most critical tests.

For the time dependent properties, creep and fatigue tests with environmental exposures are required. Particular mechanical properties required were not discussed. The tests should be conducted under actual flight load and environmental profiles. The possible use of load and temperature enhancement should be considered to accelerate the tests. All tests should be conducted at the coupon or element level to avoid costly large scale testing.

If no fatigue failure is obtained in the planned test period, a residual strength and a safety factor may be determined or the temperatures and loads may be increased to cause fatigue failure. The physical and mechanical properties and failure modes from accelerated and real time testing should be compared.

Recommendations: It was the group's suggestion that NASA should immediately select two promising materials currently available and begin the static and aging tests. It was acknowledged that the material may not be available in the future, but the methodologies developed would be. This recommendation assumes that the test techniques and results are material independent, this may not be the case.

Working Group 2

Question: How does one assess long term durability and develop appropriate accelerated test techniques?

This group was chaired by Sam Dastin of Grumman, Ben Hillberry National Research Council at NASA Langley served as the recorder. Fifteen attendees participated in this work session.

As had been emphasized in the presentations, the sensitivity of polymeric composites to a variety of load and environmental conditions, particularly in long term applications associated with durability, will require a large number of analytical and experimental programs to characterize the material behavior. Because of this complexity and the associated costs involved, careful planning and coordination of the development program will be essential. The experiences of several group members who had previously participated in similar large composite material development programs provided valuable input related to the overall management and economics of a future program.

In the context of this workshop, durability was defined as: a component satisfying the design life, or multiple thereof, when subjected to the load and environment of its intended use. For the HSCT application, required life was defined as two times design life, 100000 - 120000 hours. Expected peak temperature would be 450 degrees F, unless otherwise specified. Several major issues were identified and discussed by the group. This discussion is summarized below:

A. Analytical Modeling

Because of the large number of variables involved and material degradation in long term applications, understanding the mechanics of the material behavior and developing appropriate analytical models will be key to the success of developing and implementing high temperature polymer composites. Experimental work (test samples, techniques, environments etc.) should be focused on providing information which will assist in developing and/or verifying these analytical models. A building block approach should be used which will assist in understanding the coupling/decoupling effects of the various load and environmental parameters.

B. Materials
It is important to have pedigree materials which are well documented and verified (by the supplier). This will insure that test results are not influenced by poor material or improper processing.

C. Test Specimens
Test specimens should be of a proven design and properly prepared so as not to affect results. Factors to consider include: size, thickness, edge effects, tension/compression. Recommended test specimen sizes and geometries need to be established early in the program.

D. Testing Techniques and Equipment
Because of long term degradation enhanced by load, high temperature and environmental conditions these materials are not easy to test and will require new and difficult test techniques. Accuracy and reliability of measurement equipment and methods are critical to obtaining valid data and will require special attention. A review of available control and monitoring equipment for testing at these temperatures and moisture levels for 60000 to 120000 hours is needed.

ASTM and SACMA could assist in developing test standards to help insure valid test results. They could also assist in conducting round robin test programs.

E. Mission Profile
Mission profiles need to be defined early in the development program to provide a common basis for comparison of test results. These definitions should consider anticipated use, time to next cycle, mean and worse case conditions, ratio of supersonic to subsonic flights, and environment. Different profiles will be needed for primary structure, secondary structure, tension dominated, compression dominated, and reversed tension-compression loading conditions.

F. Economical Testing Techniques
Because of the magnitude and anticipated costs, economical testing techniques should be considered to provide more cost effective results. The following factors should be considered: multiple specimen testing, preliminary sensitivity studies, experimental design methods, reserve or extra specimens, avoid duplication of effort, one supplier of given material, one laboratory make all specimens for a given material, pool data from different laboratories, and run between laboratories round robin test programs.

G. Accelerated Testing
The development and validation of appropriate accelerated testing techniques should be accomplished early in the program to reduce costs for further testing. The development and validation of analytical models will be key to the development of accelerated testing techniques. Load truncation methods should be considered.

H. Full-Scale Structure
Full-scale structures testing should be done only for FAA approval.

I. Worldwide Technology
It is important that the program leaders and researchers be fully aware of what other countries are doing in the development of high temperature PMCs.

Recommendations: NASA should immediately fund a program to develop a series of specimen types which can be used to provide valid, meaningful data. The specimen design should be based on analysis and experimental verification. The purpose of these specimens will be to provide data that will assist in identifying and characterizing load and environmental behavior to be used in modeling. The program should begin with a thorough review of the literature for test specimens and appropriate analytical methods.

Working Group 3

Question: How does one assess the time dependent effects of a polymeric matrix composite used at high temperatures? What role does thermal aging play and how can it be modeled?

This session was chaired by Hal Brinson of the University of Texas at San Antonio. Tom Gates of NASA Langley served as the recorder. This session had eleven attendees.

The group agreed that a definition of time-dependent behavior should include such effects as aging, environment and memory. The minimum test standards necessary to establish time-dependent behavior of polymer matrix composites were determined to be:

- Establish glass transition temperature (T_g) as a function of time, moisture and fuel;
- Arrive at a means for time-temperature acceleration characterization of laminas by testing for transverse and off-axis properties. Produce short term data up to 600 hours and develop a master curve. Extrapolate to 6000 hours and experimentally verify prediction. Predict laminate results in tension and compression.
- Material aging must be assessed as a function of temperature and moisture to determine thermal stability.
- Failure and fracture of the material should be assessed in two ways: (1) Lamina transverse creep to rupture tests and (2) DCB fracture tests, da/dt, G_I, and G_{II}.
- The neat resin should be evaluated for research purposes to understand rate-dependent effects.
- Failure surfaces should be examined to determine how the failure mechanisms relate to stress and environment.

The above set of tests should be used to establish upper use limits for a given material system.

Recommendation: NASA should support efforts to establish time-dependent analytical models to predict response at 60,000 hours.

PLANNED FUTURE ACTIONS OF NASA LANGLEY

NASA's prime objective in the High Speed Research Program is to spur the technology development necessary for a commercial supersonic transport to be built in the United States. The objective of the Materials Division at NASA Langley is to promote the development, evaluation, and understanding of candidate HSR materials; and the development of mechanics-based methodology necessary to predict the deformation, strength, and life of such materials. Although the Materials Division at Langley is actively working in the advanced metallic materials and in metal matrix composites, the focus of this workshop and thus this future plan of action will focus on polymeric composites for high temperature applications.

In-House Activities

Mechanics of Materials Branch

The Mechanics of Materials Branch (MeMB) will focus on research that will eventually lead to a lifetime prediction methodology for polymeric matrix composites used at elevated temperatures. As a direct result of the workshop, several technical issues were identified and MeMB is establishing a research program to work the critical areas. This program will consist of using the most promising high temperature PMCs available today as model materials and start a combined experimental and analytical research programs. One aspect of the program will focus on the time dependent behavior. Constituent models will be developed to predict the effects of thermal aging, load, temperature and time on the composites' time dependent mechanical behavior. Another related research effort will address the thermal-mechanical fatigue behavior of the PMCs. This will define the roles that the thermal and the mechanical stresses in the constituents play in the damage initiation, accumulation and final fracture of the composite. Variables such as cyclic frequency, temperature levels, and hold times will be studied. These two programs will provide the bases for the development of an accelerated testing methodology.

Another effort of MeMB, based on the workshop suggestions, will focus on generating real-time long term durability data on the best candidate PMC, commercially available in the near future. These tests will consist of panels subjected to realistic load-temperature profiles. Two material thicknesses and load-temperature profiles representing fuselage and wing structure will be evaluated. After predetermined time increments, panels will be removed from testing, cut into several specimen geometries and tested to determine which material properties are sensitive to the long-term real time testing (eg. open hole compression, tensile strength, interlaminar toughness, etc.). These data will also serve to evaluate accelerated test procedures.

Polymeric Material Branch

In the Polymeric Materials Branch (PMB), work directed towards the development of adhesives and composites for use on high speed commercial transports involves several different area. Development and evaluation of new high temperature polymers has always been the charter of PMB, so this effort is not a new start but rather a program focus. New polymers are synthesized and characterized in neat resin forms as films and moldings and also as adhesives and composite matrices. The synthetic work primarily concerns polyimides, poly(arylene ethers) and blends of thermoplastics having high glass transition temperatures and reactive thermoplastics with thermally stable thermosetting materials. The characterization work includes the determination of various mechanical properties on neat resin, adhesive and composite specimens at elevated temperatures after thermal aging exposure. In addition, specimens are being tested after cyclic temperature exposure. In the more applied area, studies are being conducted to identify the most efficient ways of fabricating adhesive panels and composites to obtain the maximum mechanical properties. As part of this study, the molecular weight of various polymers have been controlled to lower their melt viscosity and thereby improve their compressive moldability. Many of these polymers are also end-capped to provide good melt stability. New ways to prepare adhesive tape and prepreg via slurry and powder impregnation are under study. For example, powder coated polyimide towpreg has been woven into fabric that was used to fabricate high quality composites exhibiting good mechanical properties. Some PMB polymers have shown very promising performance. As examples, a polyimide exhibited no loss in adhesive properties at 232^{o}C after aging at 232^{o}C in air for 47,000 hours. A semi-crystalline polyimide composite retained 80% of room temperature flexual strength after aging for 100 hours at 316^{o}C in air and testing at 232^{o}C. A thermoplastic polyimide toughened PMR-15 provided composites with significantly higher toughness than PMR-15 and equivalent elevated temperature mechanical properties. Future work is anticipated to show significant advancement in the development of structural resins for high speed commercial transport applications.

Contracted Activities

A significantly funded materials and structures element has been included in the Phase II HSR Program but not in Phase I. Therefore, contracted materials related research will be minimal until 1993. However, NASA is currently forming an industry team (Boeing, Douglas, etc.) that will cooperatively screen and evaluate candidate materials and share data. Tests will be conducted using common specimen geometries and testing techniques. This team activity should greatly spur the material development and screening activities, such that when money becomes available in 1993, we will know which material systems to focus upon.

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15. Supplementary Notes

16. Abstract

A workshop was held at the NASA Langley Research Center on March 14-15, 1990, to help assess the state-of-the-art in evaluating the long term durability of PMCs and to recommend future activities. Forty engineers and scientists with experience in the areas of design and evaluation of PMCs at elevated temperatures were invited to participate. The purpose of this White Paper is to briefly summarize the workshop presentations, the findings of the workshop sessions, and to outline the future plans of the Materials Division of NASA Langley Research Center.

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