Report on Thermal Cycling Testing and Bimaterial Weld Development for a Passively Actuated Materials Surveillance Test Article

Applied Materials Division

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ABSTRACT

This report describes the continuation and conclusion of a thermal cycling test program designed to evaluate a family of passively actuated material surveillance test articles. Additionally, the report details a preliminary study of bimetallic welding between 316 stainless steel and TZM (tungsten-zirconium-molybdenum) alloy. The test program was ultimately successful in demonstrating the basic viability of the passively actuated test article concept as well as specifically validating the procedures developed to size specimens to mimic the mechanical response of operating components. The report discusses and summarizes the results of the complete test program which tested samples for more than 1,000 controlled thermal cycles. The weld development program included both stir friction and electron beam joining. The stir friction welding was ultimately successful, while the electron beam welding would require additional development. Ultimately, these test programs pave the way for implementing a materials surveillance program in future molten salt reactors (MSRs). The report conclusions outline a plan for transforming the research and developed described here into a definite set of practices and procedures for a surveillance program at a future operating reactor.

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1 Introduction

This report summarizes the conclusion of a testing and development program undertaken to determine the viability of passively actuated material surveillance specimens aimed for future use in material surveillance programs in Molten Salt Reactors (MSRs). The MSR operating environment could challenge the long-term corrosion and radiation resistance of currently-qualified structural materials [1]. One way to mitigate the potentially detrimental effects of the reactor environment could be through a materials surveillance program. Such a program would monitor the current strength and reliability of component structural materials exposed to the actual, or realistically similar, reactor environmental conditions. Past work suggests that creep-fatigue damage induced by cyclic loading caused by temperature variations due to normal or unexpected operations in reactor components will be the dominant structural failure mode in operating MSRs [2]. As such, any MSR surveillance program should principally examine the effect of the reactor environment on the creep-fatigue resistance of the key structural materials. This type of program would then require a means to induce repeated, cyclic deformation on material samples, in addition to exposing those samples to the reactor environment.

One way to implement such a surveillance program would be with passively actuated surveillance specimens. Specimens of this type induce mechanical load in a test material solely through isothermal temperature variations in the sample as a whole. If placed in an operating component, samples of this type could induce cyclic mechanical load in the test material driven only by the same temperature variations that cause creep-fatigue damage in the actual component. These passive specimens would then eliminate the need to provide penetrations in the reactor coolant boundary to pass control signals and/or the driving force to the sample to induce mechanical cycling. Moreover, if the specimens can be periodically removed for inspection, for example during plant outages, then penetrations in the coolant boundary for monitoring signals could likewise be eliminated, greatly reducing the engineering burden required to implement the surveillance program in terms of component design and maintenance.

Past work [3-5] demonstrates the basic viability of a family of passively actuated surveillance specimens driven by differences in the coefficient of thermal expansion (CTE) between two materials: the test material, i.e. the qualified material used in constructing the component, and a driver material, which may not need to be qualified for use in reactor structural component, as its only function is to provide the mechanical driving force to the test material.

Three technical reports [3-5] summarize the accomplishments of this research program, which include:

1. A specimen design that can induce tensile stress on the test material using a driver material with a lower coefficient of thermal expansion. This is significant as tensile stress is often more damaging than compressive stress and austenitic stainless steels – likely materials for future MSRs – and these stainless steels have very high coefficients of thermal expansion.
2. Successful fabrication of two families of test articles with 316H stainless steel as the test material and the Ni-based Alloy 617 as the driver material. The two families of specimens are a “large” specimen with a 2 in. outer diameter and a 12 in. length and a “small” family with a 1 in. diameter and a 3 in. length. The large specimens are big enough to instrument

with strain gauges and were sized to fail in a reasonably-achievable number of controlled thermal cycles in a box furnace. The smaller specimens were sized to induce a realistic component mechanical load on the test article, which means that the predicted life of the test section is on the order of years, not months. However, the small specimens provide a test to demonstrate the fabrication and machining techniques can be used to produce a realistically-sized specimen for actual future reactor operations.

3. Both specimens require bimetallic welds between 316H and Alloy 617. The research program applied two different techniques to fabricate these welds: stir friction welding and electron beam welding. Successfully completing these welds required development work in collaboration with the welding vendors.
4. A method for sizing the specimen geometry to target a particular level of mechanical loading on the test section, given geometrical constraints on the overall test article dimensions. This sizing approach would be a critical ingredient in developing an actual material surveillance program, as it would allow the plant engineers to size the test articles to mimic the mechanical load in the actual component.
5. Methods for applying the sizing approach to size actual test articles for a component based on the design data and analysis used in developing the component design with the ASME Boiler and Pressure Vessel Code Section III, Division 5 rules. These sizing approaches are one potential means for actually implementing a materials surveillance program in a planned, future reactor.
6. A thermal cycling test program. This program cycles samples from both the large and small families in a box furnace under a controlled thermal cycle. Thus far, this test program has:
 - a. Validated the specimen sizing approach by demonstrating that the actual strain range in the large specimens, as measured via strain gauges, matches the strain range predicted by the sizing approach for the large sample.
 - b. Demonstrated that the bimetallic welds are sufficiently strong to continue to deliver mechanical load to the test section, at least over the current duration of the tests.
 - c. Generally proved the variability of the passively actuated samples and the material surveillance concept by actually fabricating and testing a number of samples under realistic thermal conditions.

This report summarizes the conclusions of this research and development program by reporting on two comparatively short-term follow-on tasks:

1. The conclusion of the thermal cycling test program. The remaining goal for continuing the thermal cycling is to induce failure in the test section of the large family of specimens, to validate the initial prediction of failure in 1,000-8,000 cycles and to demonstrate the continued integrity of the bimetallic welds. Chapter 2 describes this work.

2. The extension of the two bimetallic joining techniques to use refractory materials as the driver in the current specimen design. This then involves joining 316H stainless steel to a refractory materials, here TZM (tungsten-zirconium-molybdenum) molybdenum alloy. Chapter 3 describes this work.

Finally, Chapter 4 summarizes the conclusions of these two follow on studies and provides some suggestions for future work to fully-develop the passively actuated test articles and the corresponding methods and procedures for a complete material surveillance program.

2 Thermal Cycling Testing to Failure

2.1 Summary of the experiment and past results

As a key component of a materials surveillance technology for the nuclear reactor application, a family of passively actuated materials surveillance articles were fabricated and a long term demonstration run was started on 05/24/2021. These articles, see Figure 1, induce cyclic loading in the test section (gauge section in the middle of the samples shown in the figure) using only the cyclic temperature change. The temperature cycling is between $\sim 500^{\circ}\text{C}$ and $650+^{\circ}\text{C}$ per approximately 200 minutes, see Figure 2.

Figure 1. Various test articles without casings.

Figure 2. Temperature histories of a 1000 minute period at the beginning and near 09/07/2021. The furnace temperature is shown by the dark blue dots (data reaching 750 °C) and all other data are for the sample temperatures.

The collected strain gage readings are shown in Figure 3. One strain gage, D668, attached to T1 apparently started failing (gray line in the figure) and was replaced with the new gage, D629. As reported in the last report, as of 09/07/2021, the run accumulated approximately 480 thermal cycles.

Figure 3. Corrected strain histories of the run up to 09/07/2021.

As shown in Figure 4, two corrected strain histories during a 1000 minutes period at the beginning of the run and near 09/07/2021 are compared, indicating that most of the amplitude of the strains over the temperature cycle have reduced.

Figure 4. Corrected strain histories of a 1000 minute period at the beginning and near 09/07/2021.

This unexpected, gradual reduction in the strain range over many cycles would jeopardize the goal of the run, which is to induce failure in the test section, since the strain range delivered to the test section and measured by the strain gages should be constant during the run until the test section breaks.

Three potential explanations for the observed decrease in the stable strain range discussed in the previous report are:

1. The development of damage elsewhere in the test article, including the bimetallic welds.
2. Degradation of the strain gages themselves.
3. Loosening of the connection between the strain gage and the sample, which increased compliance in this connection, decreasing the measured strain reading (in fact, the decrease in the strain readings could be related to the buckling we observed in FY22, see next sections for details).

The first explanation can be checked at the end of the run via destructive examination of the sample.

The second explanation can be checked by checking the gages. Note that since only “active element” (shown as RA, see Figure 5(B)) and “dummy element” (RD) are exposed to the thermal cycling and only the active element experiences the mechanical stress (the dummy element is mechanically free), it can reasonably be assumed that the most damage to the strain gage must occur to RA, which should appear as a drift in the strain reading.

The third explanation was considered the most likely cause. In the last report, it was suggested that post-experiment characterization of the samples may be able to provide additional evidence supporting this theory. In addition, we were to install brand new gages purchased as spare to the specimens to check the theory.

Figure 5. (A) Typical strain gage equivalent circuit and (B) actual equivalent circuit for a KHCR strain gage with the bridge adaptor.

2.2 Finishing the tests

The goal of the run is to thermal cycle the specimens as much as possible to attempt to break the specimens. To investigate the theory (1) in the previous section, a destructive examination would be required and it must be performed at the end of the run. Theory (2) can be checked by checking the strain gage raw readings taken at the room temperature at the end of the run. Since the gauge sections are not expected to be compressed, but are expected to stretch at the end of the run, negative or unusually high values of the final strain gage readings at the room temperature may be considered as failure of the gauges. The theory (3) can be checked by installing new gages at some time before the end of the run. It would be sufficient to check this theory if we thermal-cycle the specimens with the new gages just for a few times before ending the run. Thus, the installation of the new gages is planned to be done a few weeks before the end of the run. With the objective of the run in mind, it was judged that the highest priority is not immediately investigate these hypotheses, but to continue the run as long as possible to see if any of the samples would actually break at the end of the run.

The furnace was turned off on 1/5/2022 and allowed to cool. The run contains 1211 thermal cycling. The entire histories of the sample temperatures and the furnace temperature are shown in Figure 6 and the corresponding corrected strain gage readings are shown in Figure 7.

Upon a close visual inspection of each sample revealed that the gauge sections have permanently stretched and buckled and it was apparent that the application of the proper cyclic tension to the gauge sections has become impossible. We decided to terminate the run at this point. For the

observed buckling to occur, the gauge sections must have permanently stretched, however we cannot tell whether the permanent stretch occurred in the last cool down period at the very end of the run or it took place from the repeated thermal cycling over the length of the run.

On the other hand, if the observed buckling gradually took place over the length of the run, it explains why the strain gage readings have gradually reduced for the same amount of the temperature cycling. As previously speculated, if there were mechanical failures in the samples such that the gauge sections could not undergo stress cycling (theory 1), it is believed that the permanent stretch would not have occurred. The strain gage readings at the end of the run (at the room temperature) indicate that there are no gages that have definitely failed. This fact implies theory (2), which assumes degradation of the strain gauges, may not be a dominant cause. It must be noted that because the gauge sections have buckled, even if a new strain gauge is attached and thermal cycling is applied to the sample, the sample together with the new gauge will not experience proper tensioning. Thus it is not possible to positively check theory (3), which assumes loosening of the mechanical connection between the strain gauge and the sample.

Figure 8 compares the corrected strain histories of a 1000 minute period near 09/07/2021 and at the end of the run. Almost all the samples continued to reduce the stress readings. SS1 sample shows relatively consistent stress readings throughout the run, but the identical sample, SS2, suffered the reduction of the stress readings as the other samples.

Figure 6. Entire temperature history for each thermocouple.

Figure 7. Entire histories of the corrected strain gages.

Figure 8. Corrected strain histories of a 1000 minute period near 09/07/2021, see Figure 4, and at the end of the run.

2.3 Postmortem on test samples

In order to quantify the buckling failure, various non-destructive, optical inspections were carried out.

The small sample does not show visible buckling at least visually through the openings. Since we have only one small sample in the furnace, it still is premature to conclude that the small sample design is less prone to buckling. However, it would not be surprising if the small specimen is comparatively undamaged, as it was sized to mimic realistically small component strain ranges and we had not anticipated it failing or accumulating significant damage during the thermal cycling test program.

Height measurements using an optical comparator were performed to directly measure the deflection of the gage section of the small sample through the windows, see Table 1 and Figure 10. The

dimensions indicated by red arrows were directly measured and given in Table 1. The gauge section is slightly bent (the direction not visible from the windows) by 0.0004-0.0008 inches over a 0.125 inches distance, which translates to the deflection of 0.2 to 0.4 degrees. However, such small deviations may also be attributed to the fabrication processes rather than in situ buckling.

Figure 9. SSS1, buckling is not shown at this angle.

Table 1. Height measurement data for SSS1, all numbers are in inches, see Figure 10 for details.

Axial locations (Z)	-X	+X	-Y	+Y
-0.125 in	0.0500	0.0501	0.0517	0.0486
0 in	0.0501	0.0506	0.0525	0.0482
+0.125 in	0.0494	0.0505	0.0517	0.0485

Figure 10. Cross sectional sketch of SSS1 showing how measurements were taken. The values in Table 1 are indicated in red arrows.

Figure 11. S1, showing the severest buckling at this angle.

Figure 12. Magnified view of S1 gage section using an optical comparator.

Figure 13. T1, showing the deflection of 1.65 degrees at this angle.

Figure 14. SS1, showing the deflection of 0.23 degrees at this angle.

Figure 15. SS2, showing the deflection of 1.65 degrees at this angle.

Figure 16. TS1, showing the deflection of 2.68 degrees at this angle.

Figure 17. TS2, showing the deflection of 1.19 degrees at this angle.

From these visual observations it is apparent that the buckling occurred during the run, which may have negatively affected the strain gage readings. The buckling failure might be caused by one of two mechanisms:

1. Classical “creep” buckling under elastic follow up. Creep deformation can cause thin sections can buckle under constant load or even constant strain. The load delivered to the test section by the driver is somewhere in between load and strain control (i.e. load delivered through elastic follow up). This so-called creep buckling can occur even without the development of damage in the section, i.e. only primary and secondary creep.
2. Buckling as a precursor to material failure, caused by damage-induced softening in the gauge section. The test section could fail via classical, time-independent buckling later in the thermal cycling history if the accumulation of material damage causes the time-independent stiffness of the section to diminish. Eventually, this softening could lead to buckling failure.

Even with continual visual monitoring of the test sections it would be difficult to distinguish between the two failure modes. However, classical creep buckling is more generally associated with long periods of steady load and the reverse loading applied to the specimen would tend to diminish the possibility of buckling by straightening the gauge during the tensile half of the cycle. This might suggest that mechanism #2 was the cause of the buckling deformation.

More importantly, without visual monitoring of the specimens during the cycling we cannot tell if the buckling occurred gradually over time during the standard cycles or all at once during the final cool down prior to removing the samples from the furnace. If we attribute the strain gage discrepancies to the development of buckling that suggests the buckling occurred gradually over time. However, at least one strain gauge was defective so we cannot definitively rule out strain

gauge failures as the cause of this gradual change in the strain range. Moreover, some very gradual diminishment of the tensile strain values (but not the strain range) is expected as creep and cyclic plasticity slowly centers the specimen hysteresis about zero mean stress. This process of centering the stress-strain hysteresis could contribute to the gradual development of buckling in the test section, as it means that the initial tensile-only loading on the test section gradually becomes a combination of tension and compression.

If we attribute the buckling failure in the specimen to gradual material softening, then the buckling deformation is in effect an early signal of material failure. Given the ~1,200 cycles the large specimens underwent, this places the number of cycles to failure in the range we initially predicted based on the predicted strain range (validated by the strain gauge readings) and the material average properties for 316H in the ASME Boiler and Pressure Vessel Code. However, generally speaking, buckling deformation, unless it immediately presages material failure, is not desirable as the bending in the gauge makes it difficult to relate the material deformation to uniaxial failure data. More experimental investigation is warranted, but at least these results should inform the specimen sizing procedure. Two checks could be added to the specimen sizing process to screen out designs likely to fail via buckling:

1. A creep buckling check (e.g. a large deformations analysis of the test article using the existing creep models) under the “operating” load cycles.
2. A check on time-independent buckling under any full unloading cycles, i.e. shutdowns before outages.

The second check is not fully sufficient to guard against buckling caused by damage-induced material softening, as it would use the initial, undamaged material properties. However, as described above, such a buckling failure might be considered to presage material failure. If so, any observed buckling not covered by these two buckling checks might be considered to indicate specimen failure in an actual surveillance program.

3 Bimaterial Welding

3.1 Benefits of a higher thermal expansion coefficient mismatch

For a fixed specimen geometry, the greater the mismatch in the coefficient of thermal expansion between the driver and test materials the greater the strain range delivered to the specimen. Figure 16 quantifies this effect for the small specimen geometry, keeping the material properties of the 316H test material fixed, using the mechanical properties of Alloy 617, but uniformly scaling the coefficient of thermal expansion of Alloy 617.

Figure 18. Plot illustrating how the predicted test section strain range increases as the CTE of the driver material decreases for the small specimens.

With a bigger CTE mismatch the specimens could either be sized to deliver greater load to the test section for a fixed geometry or a smaller specimen could be designed to deliver the same load. As such, the optimal specimen would use materials with the largest possible CTE mismatch.

Refractory materials have very small coefficients of thermal expansion, making them good driver materials for any of the currently-qualified reactor structural materials. Moreover, refractories are generally corrosion-resistant and are structurally strong, stiff, and creep-resistant. All of these properties mean they would be ideal driver materials for test articles designed to operate in MSR environments.

The current sample design requires two types of bimetallic welds: one completed with stir friction welding and the other with electron beam welding. Demonstrating the viability of these welds with refractory materials would then be the first step in fabricating test articles with refractory drivers.

3.2 Target materials

As the first potential driver material having a small thermal expansion coefficient, Titanium-Zirconium-Molybdenum (TZM) alloy was selected. We first targeted welding TZM to 316H, with a

secondary goal of welding TZM to Alloy 242 to assess the viability of refractory/Ni-alloy welds. However, due to availability, for this preliminary attempt, we obtained 316L material instead of 316H.

3.3 Stir friction welding

In the very first weld attempt produced only partial bonding between TZM and 316H, see Figure 16. The preliminary welding results indicated that the center area, which was least affected by the air, seemed to exhibit a relatively good bonding, which led us to use a shielding device to limit oxidation in subsequent attempts.

Figure 19. (A) Partial, but best welding between TZM and 316H. (B) Partial bonding, and (C) no bonding. Note that the welded composite bars shown in the photo underwent a quick bending test to verify the joint strength.

The shielding device was fabricated and sent to American Friction Welding (AFW) together with additional TZM and 316 rods. However, the weld trial with the nozzle and Ar cover gas showed no improvement in the weld quality, despite the presence of the inert atmosphere.

Figure 20. The nozzle (shielding device) to provide a cover gas during friction weld process to minimize the effects of oxygen in the air. The rod to be friction welded is inserted from the hole visible in the photo. 1/4 in tubing is connected to an argon gas supply.

Figure 21. Additional 10 pairs of 316 and TZM rods prepared for the 2nd friction weld development work.

Figure 22. Nozzle attached to the stationary side of the weld rod (TZM). The weld surface is visible inside the nozzle. Ar gas was supplied through the attached 1/4 in tubing.

Figure 23. Two weld samples after bending check. One sample (left) was welded in the air and another (right) was welded in the Ar gas. No differences were observed. Note that they are both broken in the TZM rods due to their brittleness, indicating partial success of the welding. The welded surface around the center of the rods show good bonding, but the area near the side of the rods shows no bonding and separates.

After an additional consideration at AFW, they performed the second weld trial with different weld parameters but without the nozzle and Ar cover gas. The new parameters aim ensure the weld cycle is completed quickly to prevent potential formation of intermetallics. A considerable improvement in the weld quality was obtained. A bending test demonstrated that the TZM rod broke first before the weld failed. This suggests that, at least at room temperature, the weld is “overmatched” by being stronger than the base material. One of the successful welded sample was sent to Argonne.

Figure 24. The second weld trial with new parameters. Note that the weld did not separate at all during a bending test and the TZM rod broke first.

Figure 25. One of the successfully welded TZM+316 rod.

3.4 Electron beam welding

To develop electron beam welding of TZM and SS316, 5 samples were fabricated and brought to an electron beam welding company, Sciaky, see Figure 16. It was possible to melt both TZM and SS316 using e-beam and fuse them together, however the fused mixture developed severe cracks, see Figure 17-Figure 20. It was also confirmed that e-beam could melt the TZM part and the resulting re-solidified TZM shows no signs of abnormal appearances, see Figure 21, implying the failure is from the mixture of TZM and SS316, which is consistent with the observation during the friction welding. Inspired by the success of the friction welding, we are planning to melt only

SS316 and see if the melted SS316 sufficiently diffuses into TZM to form bonding without melting TZM to avoid the formation of intermetallic.

The current results suggest that more testing will be required to form a successful electron beam weld between the two materials. However, such a weld is likely feasible.

Figure 26. TZM and 316 sent to Sciaky for e-beam welding test. Inside rod is TZM and outside cylinder is 316. E-beam welding was to be performed along the perimeter of the TZM rod.

Figure 27. One of the electron beam welded samples.

Figure 28. One of the electron beam welded samples.

Figure 29. One of the electron beam welded samples.

Figure 30. Cross section of one of the welded samples.

Figure 31. Electron beam melting of TZM followed by solidification. Left side is TZM (center piece) and right side is 316. E-beam was applied only on the TZM area.

4 Conclusions

This report describes the continuation and completion of thermal cycling tests on passively actuated materials surveillance samples as well as the results of a preliminary joining study examining the feasibility of joining austenitic stainless steels and Ni-based alloys to refractory metals.

The thermal cycling testing accomplished the original goals of:

1. Demonstrating the basic viability of the passively actuated test article concept
2. Validating the test article sizing approaches by measuring the strain on the test section over time and comparing those strain measurements to the modeling predictions used to size the specimens
3. Suggested the viability of the fabrication and joining techniques used to build the sample (at least for the combination of 316H and Alloy 617)
4. At least partially validated the material failure prediction used to size the samples.

The main challenges identified through this test program are:

1. Reliability issues with high temperature strain gauges. This may not be relevant to an actual *in situ* surveillance program if the specimens are only monitored during outages.
2. Buckling of the thin test section in the large samples, discussed in Chapter 2 above.

The weld study successfully produced a good stir friction joint between 316L and TZM. The initial electron beam weld study did not produce a viable joint, but additional work on this joining technology could lead to a good connection.

Overall then, the research program has demonstrated the basic viability of the surveillance concept. The logical next steps are to:

1. Extend the fabrication and joining methods to cover all likely MSR structural materials, for the test section, and refractory metals, for the driver section.
2. Demonstrate specimen operation under realistic environmental conditions, for example in exposure to molten salt or some other corrosive coolant and/or in exposure to radiation.
3. Begin standardizing the process of setting up and running a materials surveillance program, based on this technology, for future operating plants.

The successful test and development campaign described here lays the groundwork to accomplish all three of these tasks. Continued work, particularly on the first and second items, represents a logical continuation of the initial furnace test program. For Task 3, the first step would likely be writing draft standard procedures and evaluating those procedures experimentally, in conjunction with Tasks 1 and 2. However, as the MSRs come closer to market, accomplishing Task 3 will require engagement with reactor vendors, codes and standards bodies like the ASME, and the NRC to develop and adopt effective yet executable processes for establishing and running a materials surveillance program.

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Bibliography

- [1] J. Busby *et al.*, “Technical Gap Assessment for Materials and Component Integrity Issues for Molten Salt Reactors,” ORNL/SPR-2019/1089, Oak Ridge National Laboratory, Oak Ridge, TN, 2019.
- [2] Welding Research Council, “Recommended practices in elevated temperature design: a compendium of breeder reactor experiences (1970-1987): Volume II -- preliminary design and simplified methods,” 1991.
- [3] M. C. Messner and T.-L. Sham, “Preliminary Procedures and Acceptance Criteria for in-situ Structural Materials Surveillance for MSR,” ANL-ART-229, Argonne National Laboratory, Lemont, IL, 2021.
- [4] M. C. Messner, Y. Momozaki, E. Boron, and T.-L. Sham, “Initial development of an in-situ, passive material surveillance test article for monitoring high temperature reactor structural components,” ANL-ART-198, Argonne National Laboratory, Lemont, IL, 2020.
- [5] M. C. Messner, Y. Momozaki, E. Boron, G. Ye, and T.-L. Sham, “Fabrication and Testing of Two Passively Actuated Creep-Fatigue Surveillance Test Articles,” ANL-ART-228, Argonne National Laboratory, Lemont, IL, 2021.

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