INVESTIGATION OF THE RELEASE OF ZINC IN THE REACTOR SUMP AND THE BEHAVIOR OF DISSOLVED ZINC AT HOTSPOTS IN THE REACTOR CORE AFTER A LOSS OF COOLANT ACCIDENT

S. Renger, S. Alt, W. Kästner and A. Seeliger

Institute of Process Technology, Process Automation and Measuring Technology, Department of Nuclear Engineering / Soft Computing, University of Applied Sciences Zittau/Görlitz, Theodor-Körner-Allee 16, 02763 Zittau, Germany s.renger@hszg.de; s.alt@hszg.de; w.kaestner@hszg.de; a.seeliger@hszg.de

H. Kryk

Helmholtz-Zentrum Dresden-Rossendorf, Institute of Fluid Dynamics, P.O. Box 510119, 01314 Dresden, Germany h.kryk@hzdr.de

ABSTRACT

Generic experimental and methodical investigations were carried out aiming at the systematic elucidation of physico-chemical mechanisms and their influence on thermo-hydraulic processes, which can occur during the sump circulation operation after a loss-of-coolant accident (LOCA) in PWR. In such a case, borated coolant with dissolved zinc, formed by corrosion of zinc-coated containment installations, may reach core regions of higher temperature (hot spots).

Experimental studies were done at semi-technical scale focused on the influence of such zinc-containing coolants on thermo-hydraulics at heating configurations similar to those inside the PWR core. The impact of physico-chemical mechanisms on thermo-fluid-dynamical behavior of coolant inside a 3×3 configuration of heating rods, which act as fuel rod simulators with spacer segments, was determined. As a test parameter, borated coolant with dissolved zinc was used with an initial fluid temperature in the range of 45...50°C. The initial boric acid concentration in the experiments is based on the accident scenario in german PWR. In the case of a LOCA, emergency cooling water (containing 2,500 ppm boron) from two refueling water storage tanks is injected into the primary cooling circuit during LOCA.

During heat-up of zinc-containing coolant in a circuit, an increasing turbidity of the coolant fluid caused by forming colloids was observed first. This was followed by the formation of solid corrosion products consisting of zinc borates (identified by means of Raman spectroscopy [2]). In dependence of the temperatures of fluid and heatable surfaces, the solids showed various attributes concerning their mobilization potential, their density and their ability to form deposit layers. Deposits occurred at the rod surfaces as well as at the spacer segments. They effected a hindered heat transfer from the rod surfaces to the fluid, an increasing head loss at the spacers and some changes of the flow distribution. In addition, quantifications of the formed solid corrosion products including a characterization of released solid particles were done by means of microscope and particle analyzer. Subsequently, investigations were expanded considering original zinc sources (zinc-coated gratings) and a 16×16 shortened PWR fuel assembly dummy with a centered 8×8 heating rod configuration.

Achieved experimental results allow conclusions about the solubility behavior of zinc corrosion products in borated coolant as well as about the formation of solids and the consequences thereof. Beside processes of deposit layer formation and particle release, further effects like outgassing of dissolved air and local subcooled boiling phenomena were observed, which can accelerate the remobilization of formed solids and may strengthen the mentioned mechanisms.

KEYWORDS

loss of coolant accident, particle formation, corrosion, zinc release, zinc borate

1. INTRODUCTION

For the safe operation of light water reactors, the controllability of different accident scenarios must be ensured. One of these scenarios is the loss-of-coolant accident (LOCA) caused by a leakage in the primary cooling circuit. To remove decay heat from the core after a LOCA, leaking coolant will be collected in the reactor sump. The coolant will be recirculated to the reactor by residual-heat removal pumps as a part of the emergency core cooling system (ECCS). Different international investigations regarding LOCA along with the release of debris like pipeline insulation material as well as sump strainer clogging issues were performed in the past [1].

The investigations described herein concentrate on possible chemical effects during a LOCA. During the sump recirculation, leaking coolant flows over containment installations of hot-dip galvanized steel, e.g. gratings, flight of stairs, inspection platforms, room divider and support grids of sump strainers. This leads to flow-induced corrosion processes. The layer structure of the zinc-coated gratings (pure zinc as external layer, hard zinc layer, basic steel material) was determined by analyses of micrographs. The zinc layer promotes a release of soluble corrosion products in form of zinc ions, causing a change of the coolant chemistry. These corrosion effects affect german PWR in particular, because the coolant can contain up to 2500 ppm boron in form of boric acid.



Figure 1. Scheme of a LOCA scenario inside a PWR

The chemical background and the mechanisms of the formation of several zinc borates are described in detail in [2]. To estimate the temperature-dependent solubility of zinc corrosion products in boric acid containing media batch experiments were carried out [2]. The experimental results show a decreasing solubility of zinc in boric acid solutions with increasing temperature. Transferred to the LOCA scenario shown in Figure 1, the saturation concentration of zinc corrosion products in heated coolant in the core is lower in comparison to that in the coolant in the reactor sump. Hence, the decreasing solubility caused by the different concentration limits can lead to zinc-containing depositions in the core.

Within joint research projects of HSZG (University of Applied Sciences Zittau/Görlitz) and HZDR (Helmholtz-Zentrum Dresden – Rossendorf), generic experimental investigations regarding the formation

and deposition of solid corrosion products as a result of zinc corrosion processes in flows of borated coolant as well as their behavior in downstream components have been performed.

2. EXPERIMENTAL SETUP

For the investigations, a double-tracked experimental strategy was pursued. Two test facilities for experiments at semi-technical scale were built up respectively extended at the HSZG:

- "Ring Line II" (RLII), a modular test facility for investigating multiphase flow phenomena (gas-solid-liquid flows) in a cooling circuit.
- "Zittau Flow Tray" (ZFT), a sump model coupled with a partially heatable, shortened fuel assembly (FA) dummy

Furthermore, experiments at laboratory scale were carried out at the HZDR at two test facilities:

- a stirred tank reactor for clarifying basic electrochemical corrosion and precipitation mechanisms
- corrosion test facility "KorrVA" for the investigation of zinc corrosion and corrosion product deposition at hot surfaces in a single flow channel

The lab-scale experiments as well as the related facilities are further described in [2] and [5].

2.1. Test Rig RLII with 3×3 heating rod configuration

Ring Line II, an existing test facility for the investigation of different multiphase flow phenomena regarding LOCA, was extended by a 3×3 heating rod configuration. Each heating rod has a controllable power of 1.160 kW maximum. Its cladding tube is made of zircaloy. Figure 2 shows the scheme of test rig RLII (a) and its observation part (b), containing the heating rods (c) with number and position of the TC.



Figure 2. Scheme of the test facility "Ring line II" (a), equipped with an observation part (b) and a 3×3 configuration of heating rods with integrated thermocouples (c)

The filling volume of the cooling loop including water reservoir, pipes and observation part is up to 320 L. For realizing a realistic flow profile inside the rod configuration, at least one original spacer segment is used. Some rods contain thermocouples (TC) at several height positions (see Figure 2 (c)). The rod configuration is placed in a separate metal housing with the dimensions of 50×50 mm in cross section, which contains several observation windows, made of heat- and pressure-resistant borosilicate glass. Its instrumentation consists of a magnetic flow meter (MFM) for volume flow measurement and control, a conductivity probe (CP), some TC for local temperature measurements in the coolant as well as differential pressure gauges. For the online observation of microscopic effects on heatable surfaces, a

digital microscope is mounted at an observation window. At inlet and outlet of the heating rod section (heat channel, HC), the turbidity of the coolant is measured by thrubeam laser sensors.

Experimental conditions are oriented towards postulated LOCA scenarios in a generic german PWR. Several experiments were done by varying parameters like the heat output of the heating rods, the initial zinc concentration in the coolant, the presence of a zinc source and the number of spacer segments. In addition to this, the positions of the spacer were varied as follows:

- flow-sided, at the lower end of the rod configuration
- inside a window area, allowing a full observation of the deposition process (see Figure 2 (b))
- between the upper two observation windows, allowing measurement of head loss
- at the height position of the thermocouples

For most experiments at the RLII the fluid was prepared in a separate test facility. Therefore, pure zinc granules were placed under a water jet with a temperature of 45 °C until the maximum solubility was reached. In addition, some experiments were performed with the fluid of the test facility ZFT. The starting pH value and Zn-concentration were in the range of 6.61 to 6.78 respectively 78 up to 93 mg/l. The inflow temperature was in the range of 60-73 at the inlet and 69-88 at the outlet of the test section depending on the heating power.

Furthermore, the influences of fibrous insulation material as core debris on the deposition of zinc borate and on the head loss were analyzed in one experiment. Results can be found in [3].

2.2. Test Rig ZFT with partly heatable FA dummy

The ZFT is a scaled sump model of german NPP with the dimensions of $3 \times 1 \times 6$ m (H×W×L). For the investigation of downstream effects, different FA dummies (single or as a cluster) can be connected downstream the sump suction area of the ZFT.

The following properties can be varied for specifying the experimental conditions:

- volume of boric acid solution: 8 17 m³ (boron concentration: 2200 ppm)
- positions of zinc-coated gratings (ZCG) inside the coolant circuit (below leakage jet, immersed...)
- type of leakage jet (compact, scattered)
- presence of sump strainers and/or debris materials, e.g. fibrous insulation material (experiments with insulation material MD2 were performed in the previous project and issued in [6])

• heat output of the heating rods etc.

Figure 3 shows the positions of installed ZCG during LOCA experiments.



Figure 3. Positions of the ZCG in the ZFT in side and top view (a), ZCG mounted below leaking jet position in the ZFT (b)

The instrumentation consists of

- MFM for volume flow,
- TC for measurement of fluid temperature at different position in the test rig,
- TC for measurement of the temperature in the heating rods,
- Thrubeam laser sensors for turbidity measurement and
- CP at in- and outlet of the heating section.

The scheme of the ZFT with the partially heatable FA dummy is shown in Figure 4.



Figure 4. Scheme of the ZFT with partially heatable FA dummy

In the section downstream the sump suction area of the ZFT, a single shortened 16×16 FA dummy with two spacers is embedded. Figure 5 shows the arrangement of the heating rods and the TC. The center (8×8) of the FA dummy contains 56 heating rods and 8 control rod guide tubes. Five heating rods are equipped with two internal TC each.



Figure 5. Position scheme of the heating rods, in part equipped with TC (a), TC placed in coolant between the tubes (b)

3. EXPERIMENTAL RESULTS

3.1. Experimental results at RLII with 3×3 heating rod configuration

The following boundary conditions were kept identical for all experiments:

- initial fluid temperature: $T_{\text{fluid}} \approx 45 \text{ }^{\circ}\text{C}$
- initial concentrations: 13 g/L boric acid, 0.5 ppm Li
- volume flow: $\dot{V} = 0.202 \text{ m}^3/\text{h}$

Within the series of experiments, the power of heating rods, the number and position of spacers and the initial concentration of dissolved zinc were varied. In some experiments (namely RLII-HZ9-V01 to V04), a zinc source consisting of pure zinc granules was placed in the water reservoir of the RLII to simulate a permanent contact between coolant and the ZCG in the reactor sump. Additionally, one experiment with released insulation material was realized. Further details are documented in [4].

The zinc concentration in the coolant was calculated based on the online data of electrical conductivity. Assuming an unchanged concentration of boric acid (the mass of boron in the precipitated zinc borate can be neglected in comparison to the comparable high boric acid concentration in the fluid) as well as lithium hydroxide in the coolant, a measured conductivity value can be converted into a zinc concentration value by an equation empirically determined by the HZDR. Furthermore, coolant samples of the solution were taken periodically for chemical analyses.

Starting with initial zinc concentrations in the range of 78 to 93 mg/L, the concentrations decreased after heating-up continuously in all experiments. Figure 6 shows the courses of the zinc concentration in the coolant for selected experiments done at the RLII. The heating power in the selected experiments is listed in Table I. In V02 a zinc source in the water reservoir and a fine filter behind the heating section were installed during the whole experiment.

Table I. Heating power in the selected experiments at the RLII

experiment no.	V02	V07	V10	V14
heating power [W/cm]	6,47	6,56	5,36	2,71

The decline of the concentration values over the time indicates the formation of insoluble and/or hardly soluble zinc compounds.



Figure 6. Zinc concentration courses in experiments done at RLII

For experiments with $T_{fluid} > 90$ °C (e.g. V02), the final concentrations lied between 17 to 20 mg/L. The zinc concentration decreased to 40 % of the starting concentration for experiments with a fluid temperature $T_{fluid} < 80$ °C. In experiments with fluid temperatures $T_{fluid} > 80$ °C, up to 70 – 85 % of the dissolved zinc ions at the beginning were turned into zinc borate. To analyze mobilization effects and transport of solid particles out of the HC, the [0;1]-standardized values of fluid turbidity at in- and outlet were analyzed. Figure 7 shows the turbidity in dependence of the local fluid temperature at the outlet of the HC for the selected experiments.



Figure 7. Turbidity and local fluid temperature at the HC outlet for selected experiments

The measurements show that for most experiments the turbidity starts to increases at $T_{Fluid} \approx 63$ °C, hence the flocculation starts at this temperature range. In single experiments, the increase could be observed at $T_{Fluid} \approx 58$ °C (V14).

Figure 8 shows the courses of head loss (P101), turbidity at the outlet (turbidity_out), pump control current (PuCtrl) and the heating power of the tube (P_tube) of experiment RLII-HZ9-V20. In this experiment, heating power was turned off several times to investigate the dissolving behavior of formed zinc borate. The pump was automatically controlled for ensuring a constant flow rate in the HC.



Figure 8. Pump control current, turbidity, head loss and heating power in experiment no. RLII-HZ9-V20

The values indicate the relation between turbidity of the coolant and head loss as well as the influence of the forming deposits respectively head loss on the pump control. The comparably high zinc concentration

of 129.4 mg/L at the beginning of the experiment was approximated to be near the concentration limit of zinc for the initial fluid temperature (saturation). During pre-heating of the coolant, first precipitations of zinc borate could be observed in the coolant. In the early stage of the experiment i.e. at low heating power, easily mobilizable flocculent corrosion products were formed and accumulated at the spacer and the heating rod surfaces in the HC. During the heating periods (T \approx {0; 25; 50; 120} h), increasing turbidity was measured at the outlet of the HC due to newly released particles of zinc borate. This was often followed by a decrease of the head loss. Released material was transported with the coolant through the circuit back to the inlet of the HC, which often led to new deposits at the rod surfaces and the spacer segments. Upstream of the spacer segments, air pockets were observed which were caused by degassing effects. After the test, sediments of zinc corrosion products were found in the water reservoir and other components of the RLII (Figure 9).



Figure 9. Sediments of zinc corrosion products at the outlet of the HC (a) and in the water reservoir (b) in experiment no. RLII-HZ9-V20

Until the end of the experiment, corrosion products accumulated consecutively at the surface of the heating rods as well as at the spacer.

The experiments done at the RLII can be summarized as follows, assuming a coolant with an initial zinc concentration up to 90 mg/L: During the heating-up of the zinc-containing coolant, two formation mechanisms were observed. At first, flocculent particles in the fluid were formed up to a temperature of $T_{fluid} = 60 - 70$ °C (Figure 10).



Figure 10. Development of a deposit layer on heating rod surface for local fluid temperature range of 60-70 °C (exp.no. RLII-HZ9-V08, starting concentrations: Zn 86.2 mg/L; boric acid: 13 g/L)

Heatable and nonheatable parts of the HC were covered with these corrosion products. These flocculent particles adhere barely at surfaces and can be mobilized by slight flow instabilities. As a result, flakes partially went into dissolution or were detached and entrained by the coolant flow. These particles

accumulate at flow bottlenecks like spacers or pipe knees. The releasing process can be verified by the measured turbidity of the coolant at the outlet of the HC. Flakes that remain on a heatable surface for many hours often compacted to a dense pasty substance.

By heating up the coolant to $T_{fluid} = 80^{\circ}$ C, solid layer-forming deposits occur at hot surfaces (Figure 11). These layers are much more compact and not remobilizable anymore. During the growth of the layer thickness, solid zinc borate particles in form of spallings occur, which are released in the coolant circuit and sediment later at bottlenecks as well.



Figure 11. Development of a deposit layer on heating rod surface for local fluid temperature range of 70-80 °C (exp.no. RLII-HZ9-V08, starting concentrations: Zn 86.2 mg/l; boric acid: 13 g/l)

The investigations done at the 3×3 heating rod configuration confirm the previously analyzed deposition processes done at lab-scale in the test rig "KorrVA" by the HZDR [5]. Analyses by means of Raman spectroscopy [2] show that the physical and chemical characteristics of the occurring zinc compounds depend on the temperature at which they form.

3.2. Experimental results at ZFT

The following boundary conditions were kept identical in all experiments at the ZFT:

- initial coolant temperature: $T_{fluid} = ca. 45 \text{ }^{\circ}C$
- boric acid concentration: 13 g/l
- volume flow:
- volume flow in FA dummy: $\dot{V}_{EA} = 5.13 \text{ m}^3/\text{h}$ (v = 5 cm/s between heating rods)
- flow direction in FA dummy: from bottom to top
- water level (volume) in ZFT: $h = 1.86 \text{ m} (V = 11.16 \text{ m}^3)$, in V07/08: $h = 1.92 \text{ m} (V = 11.52 \text{ m}^3)$

 $\dot{V}_{total} = 25.11 \text{ m}^{3}/\text{h}$

• position of leakage jet: in 5.50 m distance to sump strainer

At the test rig ZFT, experiments with the following main goals were realized:

- 1. zinc release experiments (considers only zinc sources)
- 2. zinc release and deposition experiments (considers zinc sources and sinks)
- 3. test of preventive measurements

For testing prevention measurements, coolant chemistry was changed by increasing the concentration of lithium hydroxide (LiOH), which leads to a higher pH value at the start at the experiment. Parameters of the experiments done at the ZFT are listed in Table II.

experiment no.	ZCG surface near water jet [m ²]	ZCG surface near strainer [m ²]	heating power [W/cm]	pH-Value (start)	pH-Value (end)	Li conc. [ppm]
ZFT-BE-V01	5.886	2.542	2.79	4.97	6.56	0.05
ZFT-BE-V02	5.886	2.542	5.54-3.02	6.58	6.56	0.05
ZFT-BE-V03	5.886	1.651	4.74	5.46	6.52	0.05
ZFT-BE-V04	5.886	1.651	0	7.15	7.17	38
ZFT-BE-V05	5.886	1.651	4.66	7.14	7.13	38
ZFT-BE-V06	5.886	1.651	4.66-5.17	6.65	6.89	15
ZFT-BE-V07	5.886	2.542	0	5.99	6.71	0.05
ZFT-BE-V08	5.886	2.542	4.66-5.17	5.36	6.60	0.05

Table II. Parameters of the experiments at the ZFT

Figure 12 documents the development of zinc borate deposit on heating rods during the experiment no. ZFT-BE-V03. The image series illustrates the growth of layers between two heating rods located in the area below the upper spacer. After 90 h experimental time, the deposit layers started to conjoin. 25 h later, the gap between the two rods in the observed area was completely clogged. Entrained air bubbles were also visible.



Figure 12. Development of a solid deposit layer between the heating rods upstream of the spacer (experiment no. ZFT-BE-V03)

The deposition led to a significant increase of the coolant temperatures in the center of the FA dummy as well as to an increase of the internal temperature of the heating rods in spite of a constant heating power. Figure 13 shows the coolant temperatures between the rods below the upper spacer for experiment no. ZFT-BE-V03 (for TC positions see Figure 5b).



Figure 13. Coolant temperatures between the heated rods during the first 6 days of the experiment no. ZFT-BE-V08 (TL1-TL6: from outside to the inside of the FA dummy)

The coolant temperatures at the TC positions near the center of the FA dummy started to increase significantly after t= 70..80 h. The comparison with the development of the deposit layer in Figure 12 shows good accordance. Coolant temperatures $T_{fluid} > 100$ °C implied an isolation of the TC from the coolant, caused by increasing deposits of zinc compounds.

Figure 14 shows the temperatures between (in the coolant) and inside heating rods during the comparable experiment no. ZFT-BE-V02, showing another phenomena: The measured values showed an oscillation of the temperatures inside the FA dummy, caused by the blockage in its center.



Figure 14. Oscillating coolant temperatures (TLx)/internal rod temperatures (TSxx) and total heat output (JSGES) in experiment no. ZFT-BE-V02

Oscillating coolant temperatures were also detected outside of the heatable dummy core (see graph for TL1 in Figure 14). Hence, the blockage below the upper spacer caused a change of the coolant flow profile in the FA dummy. The oscillations itself imply a periodic change of cooling and dryout in the measuring region. During this period of the experiment, the overheating protection function triggered 3 times and switched off the heat output (see graph for JSGES in Figure 14). The course of internal rod temperatures lead to the conclusion, that the coolant has not full contact with the heating rod surface anymore and the heat dissipation is therefore hindered.

After the experiments, non-heatable tubes in the external area were removed to inspect the formed corrosion products on the heating rods. Figure 15 shows the full blockage in the center of the FA dummy. All deposit layers always increase in thickness along the flow direction. Near upper spacer, the flow path in the center is completely blocked.



Figure 15. Layers of zinc corrosion products on heating rod surfaces of the FA dummy after experiment no. ZSW-BE-V02 (boric acid: 13g/l; Li: 0.5 ppm)

The layers showed a slight brownish discoloration, a sign that they contain traces of iron oxide, which means that the layer of hard zinc (alloy of zinc and iron) of at least one ZCG was exposed to the coolant.

In further experiments, the impact of alkalization by enhanced lithium hydroxide concentrations on the release of zinc ions from the ZCG and the formation of corrosion products (zinc compounds) in the core was analyzed. Figure 16 shows the results of the experiments ZFT-BE-V06 and ZFT-BE-V05 (for experimental conditions see Table II). These changes of coolant chemistry led to lower deposits at the heatable surfaces. There were differences in coloration and consistency of the deposits, which means that various zinc borates were formed here in dependence of coolant chemistry and/or that ferrous sublayers of the ZCG were not exposed to the coolant yet (minimized corrosion effects).



Figure 16. Layers of zinc corrosion products on heating rod surfaces of the FA dummy after the experiment with 15 ppm (a) and 38 ppm lithium (b)

Figure 17 shows the relation between the zinc concentration caused by corrosion and the relating pH value of the coolant for experiments carried out in RLII (RLII-HZ9-Vxx) and in the ZFT (ZFT-BE-Vxx). The release rate of zinc depends on the flow profile and the presence of entrained air. Hence, most zinc ions are released at ZCG, which are located immersed near the water surface and below the leaking jet

position or located in the leaking jet itself. Additional zinc release experiments showed that zinc ions are released even when ZCG are immersed in stagnant coolant.



Figure 17. Zn concentration in coolant vs. pH (Test rig RLII and ZFT)

4. CONCLUSIONS AND OUTLOOK

Experiments at two test facilities at semi-technical scale were carried out to analyze the behavior of ZCG under LOCA conditions and their influence on the chemistry of borated coolant. Furthermore, the consequences of these changes under boundary conditions of a reactor core generating decay heating power were investigated.

The experiments lead to formation of dissolved zinc by corrosion and deposition of corrosion products at 3×3 heated zircaloy cladding tubes (test rig RLII) respectively at the 56 heated zircaloy cladding tubes of a shortened FA dummy (test rig ZFT). In both facilities, significant amounts of solid corrosion products were found in form of layer-forming depositions on hot surfaces as well as in the form of sediments in pipes, at bottlenecks and other passive components of the rig. Chemical analysis at the HZDR identified the corrosion products as several types or even combinations of zinc borates.

Alkalization as a possible preventive measure shows, that the release of zinc ions due to corrosion can be reduced by increasing the pH value of the coolant, but it cannot be fully prevented. This statement is supported by the courses of Zn concentration over experimental time.

All analyses and experiments done so far by the HSZG and the HZDR have a generic character and allow describing the phenomena, which can be caused by the release of zinc ions into borated coolant. They allow the evaluation of condition-based mechanisms of formation, transport and deposition of zinc borates, but do not allow one the following conclusions:

- time-related assignments of all parallel running, interacting mechanisms at zinc sources and zinc sinks to real conditions and running processes during a PWR LOCA,
- any kind of statements concerning possible consequences for the dissipation of decay heat in the PWR core.

For coming experiments, boundary conditions and relations according to specific LOCA incidents in a generic PWR will be considered.

NOMENCLATURE

Conductivity Probe
Emergency Core Cooling System
Fuel Assembly
Heat Channel
University of Applied Sciences Zittau/Görlitz (Hochschule Zittau/Görlitz)
Helmholtz-Zentrum Dresden – Rossendorf
Heating Power
Lithium
Loss of Coolant Accident
Nuclear Power Plant
Pressurized Water Reactor
Test rig "Ring Line II"
Thermocouple
Thermocouple position
Zinc-Coated Gratings
Test rig "Zittau Flow Tray"
Zinc

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